Indoor Air and Community Monitoring Plan

for the

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BUREAU OF EASTERN REMEDIAL ACTION

Jackson Steel Superfund Site, Mineola, New York

Prepared for:
U.S. Environmental Protection Agency
Region II
290 Broadway, New York

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January 2005

Monitoring Plan

Indoor Air and Community Monitoring Plan,

Jackson Steel Site

Mineola, Long Island,. New York

Submitted to

U.S. Environmental Protection Agency, Region 2

January 2005

CH2MHILL

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SECTION 1

1.0 Introduction

The purpose of this plan is to describe how the following will be performed at the Jackson Steel Superfund Site and , Mineola/North Hempstead, in Nassua County, New York:

1. Collect indoor air data in specified structures around the site
2. Monitor the ambient air around the site for community ptotection

This document represents an addendum to the *Quality Assurance Project Plan (QAPP) for the Remedial Investigation at the Jackson Steel Superfund Site,* which was prepared by EPA for the indoor air sampling previously conducted by EPA in these structures (October 7, 2002).

The air monitoring plan was prepared at the beginning of the remedial investigation activities in December 2000. The plan was prepared in accordance with the New York Department of Health (NYDOH) Generic Community Monitoring Plan revision 1 date June 2000. The Jackson Steel Community Monitoring Plan has been previously reviwed and approved by NYDOH and is presented in this document.

1.1 Background and History

The Jackson Steel site is an inactive manufacturing facility located in Mineola/North Hempstead, in Nassau County, New York. Degreasing solvents including tetrachloroethylene (PCE), trichloroethylene (TCE) and 1,1,1-trichloroethane (TCA) were used at the site until 1985. The site ceased activity in 1991.

The site is bordered to the northwest by First Street, to the northeast by an apartment building, to the southeast by a billiard parlor and a day care center, and to southwest by a paved parking area, and a building with law offices and a bar/restaurant.

Now a retail Store

Two soil gas surveys and four indoor air sampling events have been performed to evaluate the presence and possible migration of VOCs through the unsaturated soil from the Jackson Steel site. These studies detected elevated concentrations of volatile organic compounds (VOCs) in soil gas and in the indoor air of the day care center (Tutor Time) and billiard parlor buildings. Based on the results of the indoor air sampling events, subslab ventilation systems were installed underneath both buildings in order to reduce the indoor air concentrations to levels below the New York Department of Health (NYDOH) guidance levels. The main VOCs identified to be present in soil gas were 1,1-DCA,1,2-DCA, cis-1,2-DCE,1,1,1-TCA, TCE, and PCE.

EPA conducted a Remedial Investigation and Feasibility Study (RI/FS) at the site to assess site contamination and evaluate appropriate remedial alternatives. The human health risk assessment performed as part of this RI/FS concluded that the indoor air monitoring data indicated that noncancer risks to children were slightly higher than USEPA's target hazard index when ventilation systems (subslab and building ventilation) were in operation. Under worst-case conditions, when ventilation systems were not in operation, cancer risks to adult workers were higher than the upper bound of USEPA's target risk range.

The Record of Decision (September 2004) selected soil vapor extraction (SVE) as the remedy for lowering soil gas concentrations and indoor air concentrations in the neighboring buildings to levels below the NYDOH guidance levels. Indoor air monitoring in the neighboring buildings is planned during SVE implementations to evaluate whether the SVE system is effective and meeting this objective.

1.2 Task Description

The purpose for this task is as follows:

- Collect baseline air samples from neighboring buildings (billiard parlor, Tutor Time, Lawyer's offices, apartment complex) to characterize indoor VOC concentrations prior to startup of the SVE pilot system.
- Collect a background air sample to characterize outdoor VOC concentrations prior to startup of the SVE pilot system.
- Collect air samples from neighboring buildings (billiard parlor, Tutor Time, Lawyer's offices, apartment complex) following start up of the SVE pilot system to evaluate its effectiveness in reducing indoor VOC concentrations. Samples will be collected monthly over a six month period with one of the sampling events timed with the injection of the chemical oxidant to determine if VOCs were released into neighboring buildings during oxidant injection.
- Monitor ambient air during onsite activities in accordance with the compliance monitoring plan.

2.0 Indoor Air Monitoring Plan

The objectives for the data collection activities presented here are:

- Collect indoor air samples to measure VOC concentrations in neighboring buildings prior to start up of the SVE pilot system.
- Collect indoor air samples at the same locations to evaluate reduction in VOC concentrations over time during operation of the SVE pilot system.

Samples will be collected with the subslab ventilation systems switched off.

Can you schedule for when $ Store, offices have fewer staff present?

2.1 Sample Locations and Sample Numbering

The study area consists of the billiard parlor, Tutor Time, office building with the lawyer's offices and restaurant, and adjacent apartment complex – all of which were sampled during the previous indoor air monitoring events. The specific sampling locations will be the same as those defined during the October 2002 indoor air monitoring event, conducted by EPA. The sample locations and a location code (used to create unique sample identification numbers) are shown below in Table 1. The sampling locations also are shown in Figure 1.

One indoor air sample will be collected from each location. There are a total of 17 sampling locations; 10 locations are in Tutor Time, 2 locations are in the billiard parlor, 2 locations are in the lawyer's office building, one in the Richlee apartment complex, and one outdoor within the fenced parking are at the site. One location within Tutor Time has been designated for a field duplicate sample. An additional sample will be collected to serve as a field blank. The total number of samples is 18. The procedure for numbering samples is shown below.

TABLE 1
Proposed Indoor Air Sampling Locations
Indoor Air Monitoring Addendum, Jackson Steel Site, Mineola, Nassau County, New York

Sample Location Code	Sample Location	Comment
TT01	Learning Center, NW	
TT02	Learning Center, SW	
TT03	Village Center	
TT04	Twaddler Room	
TT05	Infant Room	
TT06	Newborn Room	
TT07	Pookie' Cafe	
TT08	Pookie' Cafe	Field Duplicate
TT09	Toddler Room	

TABLE 1
Proposed Indoor Air Sampling Locations
Indoor Air Monitoring Addendum, Jackson Steel Site, Mineola, Nassau County, New York

Sample Location Code	Sample Location	Comment
TT10	Learning Center	
TT11	Playground	
BP01	Billiard Hall behind counter	Locations still
BP02	Billiard Hall behind rear door	make sense?
LO1	Lawyer's offices front	
LO2	Lawyer's offices back	
AP	Apartment complex boiler room	
AA	Ambient air outdoor	
FB	**NONE**	Field Blank

Sample numbering will capture the following information:

- Sample location code
- Type of sample
- Unique sampling event
- Date of sample collection

There are three types of samples:

- N – normal environmental sample
- FD – field duplicate
- FB – field blank

There are seven unique sampling events – one baseline event prior to the startup of the SVE pilot system, and six monthly events following startup of the system:

- BL – baseline
- M1 through M6 – monthly sampling events following startup

Examples of the sample number ~~is~~ are presented below. This represents a normal environmental sample from the Learning Center, NW location, collected prior to startup of the SVE pilot system (baseline) on January 15, 2005:

TT01-BL-011505-N

This represents a field duplicate from the Pookie' Café location, collected during the second monthly sampling event, on March 7, 2005:

TT08-M2-030705-FD

2.2 Pre-Sampling Survey

The owners of the Tutor Time and billiards parlor buildings will be contacted 1 week before each sampling event and requested to switch off the subslab ventilation systems. The owners will be notified to switch the system back on immediately after sampling is completed. The field team will verify that the systems are switched off before beginning sampling.

A pre-sampling survey sheet will be completed for each sample collected, to identify potential sources of chlorinated VOCs and other VOCs within the structures. Each location will be surveyed before the sample is collected during each sampling event.

The survey form is presented in Attachment 1.

2.3 Sampling Activities

Upon completion of the pre-sampling survey, the field team will deploy sampling equipment. Air samples will be collected in 6-liter evacuated stainless steel Summa canisters equipped with flow regulators for 24- hour integrated sampling.

During the baseline sampling event, the field team will collect 19 canister samples (one from each designated location within Tutor Time, billiards parlor, apartment complex, and lawyer' office; one background outdoor air sample; one field duplicate sample; and one field blank sample). The results of the outdoor background air sample will be used to evaluate whether background air quality may be contributing to any measured indoor air concentrations. The field blank will be used to determine whether any contamination may have been introduced into the canisters during the process of collecting the samples.

During five of the sampling events following start-up of the SVE system, the field team will collect 15 canister samples (one from each designated location within Tutor Time and billiards parlor, one field duplicate sample, and one field blank sample). The results will be used to evaluate whether indoor air concentrations are decreasing following start-up of the SVE system.

One of the sampling events will be timed with the injection of the chemical oxidant. During this sampling events, the field team will collect 18 canister samples (one from each designated location within Tutor Time, billiards parlor, apartment complex, and lawyer' office; one field duplicate sample; and one field blank sample). The results will be used to evaluate whether injection of the chemical oxidant into the groundwater has temporarily resulted in higher indoor air concentrations due to the volatilization of VOCs from the groundwater.

All canisters will be supplied from and returned to the <INSERT NAME OF ANALYTICAL LABORATORY> for analysis. Calibrated and cleaned flow controllers also will be supplied by the analytical laboratory. A standard operating procedure for sampling using Summa canisters is presented in Attachment 2. Examples of the field sampling and chain of custody forms are presented in Attachment 3.

A summary of the field procedures to be conducted during deployment of each sample are as follows:

- Install flow controller on the canister.

- Verify pre-sampling pressure in the canister
- Create an entry for the sample on the Indoor Air Sampling Log (see Attachment 3) – note sample location, field sample identification number, canister identification number, flow controller identification number, initial canister pressure and starting date/time (flow rates do not need to be noted – flow controllers will supplied calibrated by the laboratory) be
- Complete a pre-sampling survey sheet
- Place canister and open sampling valve.
- Prepare a field blank sample. Handle in the same manner as all other samples, but do not open the sampling value for sample collection. Valve
- After 24 hours, close sampling valve and note ending date/time on the Indoor Air Sampling Log.
- Verify post-sampling pressure in the canister and note on the Indoor Air Sampling Log.
- Complete a chain-of-custody form (see Attachment 3) and package form and canisters for shipment to the analytical laboratory.

One field sampling sheet will be started for each sampling location and information regarding all samples collected at this location will be recorded on this sampling sheet. This will provide the field sampling team with easy access and reference to setting in a consistent manner the Suma canisters.

2.4 Laboratory Analysis

EPA Method TO-15 analysis will be performed on the canisters at <INSERT NAME OF ANALYTICAL LABORATORY> located in <CITY, STATE>.

The laboratory will perform TO-15 analysis in accordance with the measurement quality objectives prescribed in the Jackson Steel Site QAPP (October 7, 2002). Laboratory data delivery will adhere the EPA CLP level 3 format. The laboratory will supply certified clean Summa canisters, and cleaned and calibrated flow regulators.

The target analyte list and associated reporting limits are shown in Table 2.

TABLE 2
Proposed Target Analyte List and Reporting Limits
Indoor Air Monitoring Addendum, Jackson Steel Site, Mineola, Nassua County, New York

Compound	Reporting limit ug/m3
1,1-DCA	4
1,2-DCA	4
cis-1,2-DCE	4
1,1,1-TCA	5.5
TCE	5.4
PCE	6.8

Reporting limits are based on limits achieved during previous indoor air sampling on October 16-17 2003.

Faxed results will be requested within 5 working days of sample receipt by the laboratory. Hard copies of data packages and electronic data deliverable will be requested within fourteen days of sample receipt by the laboratory. Laboratory will report all results in ug/m3.

2.5 Data Evaluation

Analytical results will be summarized monthly in a table and provided to the EPA. Indoor air concentrations will be compared with NYSDOH indoor air criteria. A brief narrative will be provided along with the summary table, noting key observations from the pre-sampling survey forms and any deviations from this addendum. Each monthly submittal will include the results of the previous sampling events for each location. Validation of the results is not planned as they will not be used for the purpose of determining human health risks.

SECTION 3

3.0 Community Air Monitoring Plan

Real-time air monitoring for volatile organic compounds (VOCs) will be conducted during tasks involving intrusive activities (e.g., boring, well installation). The monitoring program will be described in detail within the Site-Specific Health and Safety Plan (HASP) and is outlined below. The air monitoring will be conducted for both worker and community safety and will be associated with exposure action levels, which if exceeded, will trigger the need for corrective action. The exposure action levels will be used to ensure that potential community exposure (at perimeter boundary) to airborne contaminants are maintained at levels no greater than one order-of-magnitude less than occupational exposure limits. Because the developed action levels are based on existing site-specific contaminant data (types of contaminants, concentrations, media, exposure limits) and instrument responsiveness, these action levels are more conservative than the levels in the NYDOH Generic Community Air Monitoring Plan. The air monitoring and associated action levels will be as follows:

Table 3

Community Air Monitoring and Action Levels

Condition	Location	Action
Worker breathing zone levels > 1 ppm	Work area	Begin periodic perimeter monitoring
Perimeter levels are 1 to 5 ppm	Perimeter	Perform continual perimeter monitoring; Implement corrective actions
Perimeter levels are equal to or greater than 5 ppm.	Perimeter	Cease operations until corrective actions maintain levels below 5 ppm

Levels are sustained readings

Corrective actions could include: temporarily suspending advancement into subsurface and allowing time to passively vent; providing mechanical ventilation where feasible; relocating boring; rapid containerization of cuttings; application of foam barrier; conducting work at night; suspending operations; etc.

Air monitoring will be conducted with a MiniRae (PID) or equivalent, equipped with a 10. 6 eV lamp and calibrated to 100 ppm isobutylene. Readings from the instrument will be recorded as they are made by field staff while onsite. Continuous record of air levels is not planned.

Think about this — look for periodicity that will be proposed

also, suggest 11.7 eV lamp during Chemox

SECTION 4

4.0 Project Schedule

The baseline sampling event is scheduled to occur <INSERT DATE>. Follow up sampling events will be initiated one month following start up of the SVE pilot system. It is anticipated that all the field work can be completed in 2 working days for each sampling event. Laboratory analyses will be completed in approximately two weeks following receipt of samples.

Ambient air monitoring will be performed with the start of intrusive activities at the site.

Figures

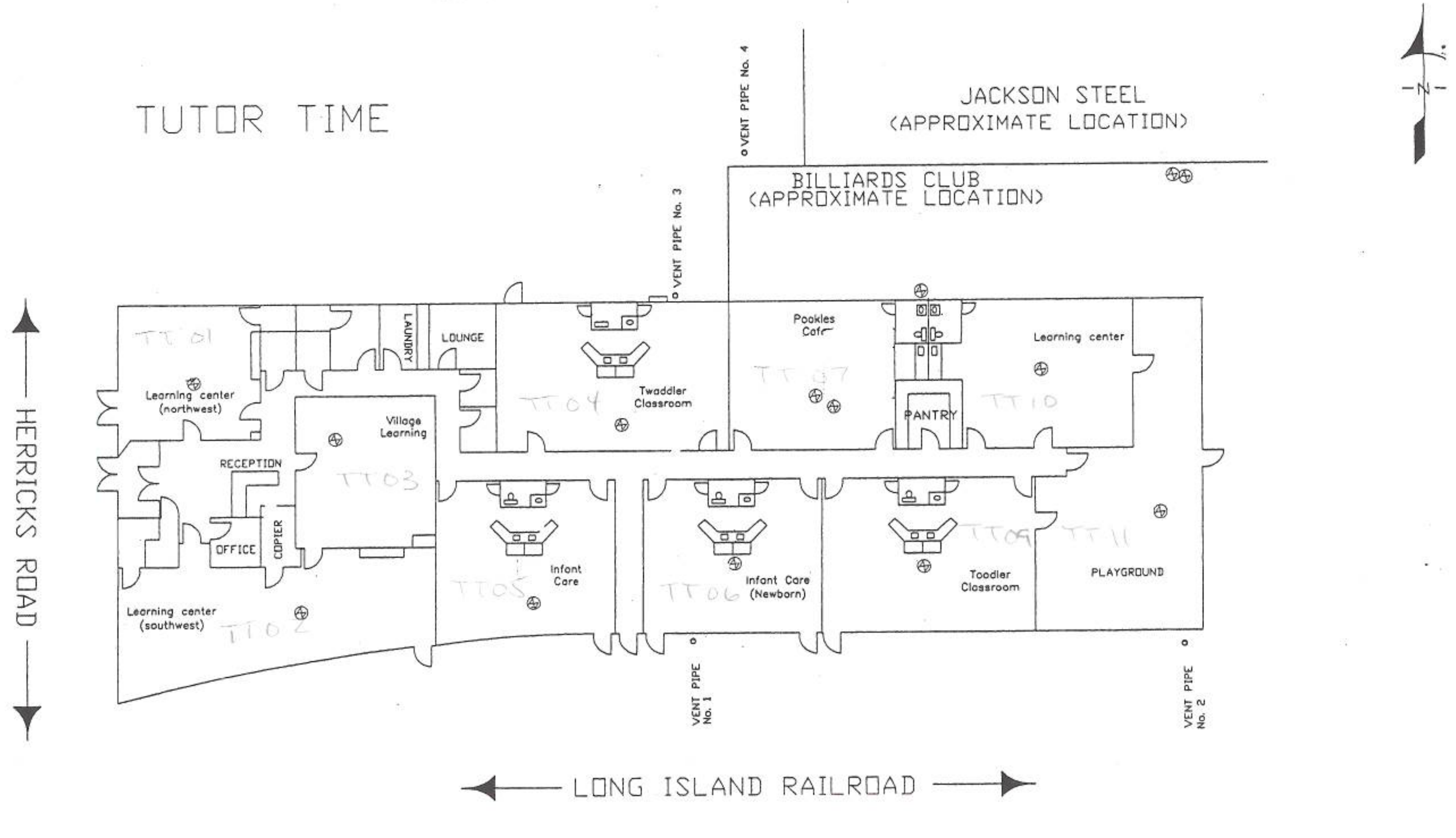


Figure 1
Jackson Steel
Proposed Sampling Locations in
Tutor Time and Billiards Parlor Buildings

Adapted from EPA Report on October 16-17 Air Sampling Results

TT08- Dup of TT07

Attachments

ATTACHMENT 1

Pre-Sampling Survey Form

INDOOR AIR QUALITY BUILDING SURVEY

Occupant/Building Name:________________________ Date:______________

Address:____

Completed by:______________________

Canister #______________________ Time Started:__________ Time Stopped:__________
Location:____
Canister #______________________ Time Started:__________ Time Stopped:__________
Location:____

Building type: residential/office/commercial/industrial

Basement size:______________ft3

Number of floors
below grade: __________ (full basement/crawl space/slab)
at or above grade:__________

Foundation type: poured concrete (over gravel)/ cinder blocks/earthen/
Other (specify) ______________________

Building occupants: Children under age 13 ____ Children age 13 - 18 ___ Adults ____

OUTSIDE SOURCES	Yes	No	Comments/Locations
Garbage dumpsters			
Heavy motor vehicle traffic			
Construction activities			
Nearby industries (identify)			
UST/AST (gasoline, heating fuel)			
BASEMENT SURVEY			
Wall construction (cinder block, poured concrete,sheet rock, paneling, etc.)			type: **condition:**
Floor Construction (earthen, slab, floating, etc)			type: **condition:**
Number of windows present on each wall and size			North: East: South: West:
Was basement painted recently? oil-base or latex paints			date: type of paint:
Sump present (PID/FID/CGI#s?)			

	YES	NO	
Location of sump			
New flooring in basement?(list type - carpet, tile)			
using glue			
New furniture added to basement			type: date:
Staining on floors/walls			
Moisture visually present in the basement			
Pipes running through walls, floor (conduits-describe, give FID/PID/CGS readings)			
Odors detected by inspector			
Basement used as living space			
Time occupants spend in basement (hours/day/per person)			
Items stored in basement: solvents			
gasoline			
paint/thinners			
polishes/waxes			
insecticides			
kerosene			
household cleaning products			
mothballs			
other items?			

NOTES: ____

____

____

FIRST FLOOR SURVEY

Wall construction (cinder block, sheet rock, paneling, etc)			type: condition:
Was painting done recently? oil-base or latex?			date: type of paint:
New flooring on 1st floor? (list type - carpet, tile)			
using glue			

	YES	NO	
New furniture added to 1st floor? (list type - carpet. tile)			type: date:
Staining on floors/walls			
Pipes running through walls, floor (describe)			
Odors detected by inspector			
Items stored on this floor			
solvents			
gasoline			
paint/thinners			
polishes/waxes			
insecticides			
kerosene			
household cleaning products			
mothballs			
other items?			

NOTES:____

____

____

PERSONAL ACTIVITIES

Does anyone in the building smoke?			
approx. number of tobacco products per day, per person			
Does anyone dry-clean their clothes?			
List hobbies of Residents			
Any house pets?			

MISCELLANEOUS

Have the occupants ever noticed unusual odors inside or outside the building ?			describe: location:
Known spill outside or inside building (Specify location)			
Type of heating used in building oil -			
natural gas			
kerosene			

electric					
other (specify)					
Is the heating unit properly vented?					
Water damage or standing water in building (historic or current)					
Fire damage to building			date:		
Pest control applications			date:		
FIELD SCREENING RESULTS	FID	PID	CGI	CO2	Rel. Hum
Basement					
First Floor					
Additional Floors					
Other (specify)____________________					
NOTES:					

NOTES:

ATTACHMENT 2

Standard Operating Procedure for Summa Canister Sampling

TO-14/15 (SUMMA® canister) Integrated Ambient or Indoor Air Sampling Method for Trace VOCs

1 Scope and Application

1.1 This sampling method describes the procedure for collecting ambient air samples for targeted volatile organic compounds. The method presented here is based on 'clean' sampling techniques. The requirements of 'clean' sampling dictate sampling and sample handling be by trained personnel.

2 Summary of Method

2.1 A sample of ambient air is withdrawn, using clean technique, from into a certified clean and evacuated SUMMA® canister by virtue of a certified clean flow controller. Sample collection can be integrated over time by adjusting the flow controller. Sample periods as short as 10 minutes to as long as 24 hours can be achieved based on the size of canister used and the sampling rate selected.

3 Apparatus and Materials

3.1 Canister, SUMMA® polished, certified clean, evacuated, and wrapped for shipping.

3.2 Flow controller, certified clean, set at desired sampling rate, and wrapped for shipping.

3.3 Shipping container, suitable for protection and canister during shipping.

3.4 Wrenches and screw driver (clean and free of contaminants), various sizes as needed for connecting fittings and making adjustment to the flow controller

3.5 Bubble flow meter or equivalent (clean and free of contaminants), used in the adjustment of the flow controller

3.6 Negative Pressure Gauge, oil free and clean, either installed on the canister or used externally to check canister pressure.

3.7 Zero air purge gas (UHP) and regulator to purge and clean flow controller between uses.

4 Procedure

4.1 'Clean' sampling protocols must be followed at times when handling and collecting samples. This requires care in the shipping, storage, and use of sampling equipment. Cleanliness of personnel who come in contact with the sampling equipment, no smoking, eating, or drinking, no perfumes, deodorants, clean clothing (not dry cleaned) and clean overgarments.

4.2 Inspect the canister for damage. Do not use a canister that has visible damage.

4.3 Verify that the vacuum pressure of the canister is equal to that indicated on the laboratory's evacuation tag. Do not use a canister that has leaked.

4.1.3Remove the protective cap from the valve on the canister.

4.2.3If using an external gauge, attach the gauge to the canister and open the valve. After taking the reading, close the canister and remove the gauge.

4.4 Verify that the flow controller is set at the desired sampling rate. If not pre-set, see the appropriate SOP for flow controller adjustment.

4.5 In the field log record the canister ID, flow controller ID, initial vacuum, desired flowrate, sample location information, and all other information pertinent to the sampling effort.

4.6 Connect the flow controller to the canister. The flow controller fitting denoted "LP" or "OUT" is

connected to the canister. Tighten the fitting as to be leak free but <u>do not over tighten</u>. (A ¼ turn past snug is usually enough.)

4.7 Remove all work articles from the sampling area.

4.8 To begin sampling, slowly open the canister valve one full turn.

4.9 For canisters with built in pressure gauges, monitor the vacuum pressure change several times during the course of the selected sample period to ensure the canister is filling at the desired rate.

4.10 At the end of the sample period, close the canister valve finger tight.

4.11 Remove the flow controller and replace the protective cap on the canister valve fitting.

4.12 If using an external vacuum gauge, re-attach it, open the canister valve, and record the pressure. Then close the valve, remove the vacuum gauge, and replace the protective cap.

4.13 If the flow controller is going to be used for more than one sample collection, be sure to purge it between uses. To do this, attach the flow controller to a vacuum source and draw clean air or gas (UHP) through it for several minutes before attaching it to the canister.

4.14 Fill out all appropriate documentation (chain of custody, sample tags) and return canisters and all equipment to the laboratory in the shipping containers provided.

4.15 When packing the canisters for shipment, verify that the valve (just past finger tight) and valve caps are snug (1/4 turn past finger tight), and use sufficient clean packing to prevent the valves from rubbing against any hard surfaces.

4.16 Please do not place sticky labels or tape on any surface of the canister!

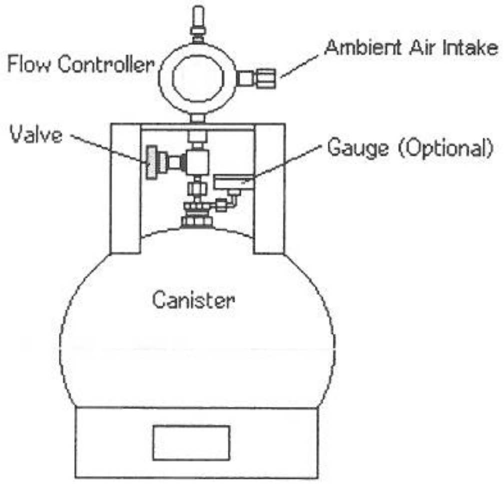
5 Quality Control

5.1 Canister supplied by the laboratory must follow the performance criteria and quality assurance prescribe in US EPA Method TO-14/15 for canister cleaning, certification of cleanliness, and leak checking. Standard operating procedures are required.

5.2 Flow controllers supplied by the laboratory must follow the performance criteria and quality assurance prescribed in US EPA Method TO-14/15 for flow controller cleaning and adjustment. Standard operating procedures are required.

FIGURE 1

Assembled Canister Sampler for Integrated Sample Collection



ATTACHMENT 3

Field Sampling Forms

Indoor Air Sampling Log (Summa Canister)

Sheet 1 of __

Project Information

Project Name:

Project #:

By:

Sample Location:

Sample Location Diagram

Date	Sample Number	Field ID	Canister ID	Flow Controller ID	Initial Canister Pressure ("Hg)	Start Date & Time	End Date & Time	Final Pressure ("Hg)	Final Flow Controller Rate (ml/min)	To	Comment

(Continued on next page)

Sample Location Diagram

N

Note:

Draw in outline the structure's foundation and interior walls, identify rooms, and note other defining features. Show location of canister relative to physical objects, etc.

Other Observations and Comments (note any unique circumstances):

SAMPLE **CHAIN OF CUSTODY RECORD**

Project/Contact Information	
Project #	
Project Name	
Report Copy to	
Company Name/Contact	

THIS AREA FOR LAB USE ONLY		
Lab #	Pg 1	of 1
Lab PM	Custody Review	
Log In	LIMS Verification	
pH	Cust Seals Y N Ice Y N	
QC Level 1 2 3		
Cooler Temperature		

Sampling		Type		Matrix					Total Number of Containers	Requested Analysis													
										TO-15 SIM													
										Preservative													
Date	Start Time	Comp	Grab	Water	Soil	Air	Client Sample ID (9 Characters Max)	LAB QC														Alternate Description	Lab ID

Sampled By	**Relinquished By**	
Received By	**Relinquished By**	
Received By	**Relinquished By**	
Received By	**Date/Time**	

Special Instructions TO-15 SIM mode for VC, PCE, TCE, and cis-1,2-DCE. The MRL will be 0.025 µg/m3, except for TCE, which will be 0.016 µg/m3

ATTACHMENT 3

Quality Assurance Project Plan October 7, 2002

QUALITY ASSURANCE PROJECT PLAN (QAPP)

FOR THE

REMEDIAL INVESTIGATION

AT THE

JACKSON STEEL SITE

MINEOLA, NASSAU COUNTY, NEW YORK

Project Officer's Signature: Diane Salkie Date: 10/7/02

Project Officer's Name: Diane Salkie, Environmental Scientist

Project Quality Assurance Officer's Signature: Pat Sheridan Date: 10/23/02

Project Quality Assurance Officer's Name: Pat Sheridan, QA Officer

Date Prepared: October 07, 2002

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Appendix B - Eastern Research Group (ERG). March 2000. *Support for NMOC/SNMOC, UATMP and PAMS Networks. Contract No. 68-D-99-007. Quality Assurance Project Plan*. Morrisville, North Carolina

Appendix C - U.S. Environmental Protection Agency (EPA). January 1999. Compendium Method *TO-15: Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specialty-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS)* from the *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air.* Second Edition. Center for Environmental Research Information. Office of Research and Development. Cincinnati, OH

Appendix D - U.S. EPA. July 1995. *Environmental Response Team (ERT) Standard Operating Procedure (SOP) #1704: SUMMA Canister Sampling.*

Appendix E - U.S. EPA. September 1994. *Environmental Response Team (ERT) Standard Operating Procedure (SOP) #170: Sample Documentation.*

Appendix F- ERG's Method Detection List

Appendix G - Example *Questionnaire*, Example *Canister Field Data Sheet* and Example *Chain of Custody* record

Appendix H - Resident Instructions

1.0 Project Description

1.1 Problem Definition/Background

The Jackson Steel site (site) is an inactive "roll form metal shapes" manufacturing facility located in Mineola/North Hempstead, Nassau County, New York. The beginning of the site's operation is unknown, however, a form/application was submitted to the Nassau County Health Department in October, 1977. Jackson Steel ceased operations in April of 1991. Degreasers including tetrachloroethylene (PCE), trichloroethylene (TCE) and 1,1,1-trichloroethane (TCA) were used at the site until March 1985. Degreasing sludge was stored in drums on the paved area located to the southwest of the building. The site property is bordered to the northwest by First Street, to the northeast by an apartment building, to the southeast by a billiard parlor and an electronics store, and to the southwest by a paved parking area, law offices and a bar/restaurant. The site is located on the edge of a mixed-use area, with commercial and industrial properties located to the south and west and residential properties located to the north and east.

The Nassau County Department of Health conducted numerous inspections of the Jackson Steel site between 1979 and 1996. An improper spill control at the waste storage area was noted in 1981 during the period when degreasers were used on site. A Limited Phase II Assessment was performed in December of 1991. During the inspection, a third dry well was observed in the loading dock of the building. Soil samples collected beneath the dry wells indicated presence of PCE, TCE, 1,1,1-TCA, 1,2-dichloroethylene (DCE) and 1,1-dichloroethane (DCA) at depths up to 17 feet below ground surface.

A reconnaissance of the site was conducted in November 1992 as part of an Environmental Site Investigation (ESI). Numerous stained areas and puddles were observed in the building. Drums containing petroleum products were stored in the building and on the paved area. The dry wells were partially filled with liquid and a 275-gallon degreasing tank was observed "in close proximity" to the dry well located in the loading dock. Between December 1992 and February 1993, nine ground water samples and numerous samples were collected in, around and below the dry wells. The analytical results indicated PCE, TCE, 1,1,1-TCA and 1,2-DCE at depths up to 40 feet below the ground level and PCE, TCE, 1,1,1-TCA, 1,2-DCE and 1,1-DCA in ground water collected from monitoring wells located downgradient of the dry wells.

The ESI report suggests that a release of PCE, TCE, and 1,1,1-TCA to ground water has occurred from the site. Drinking water within 4 miles of the site is derived from public/municipal supply wells screened in the Upper Glacial, Magothy and Lloyd Aquifers. The nearest well drawing from the aquifer of concern is located approximately 3100 feet south and side-gradient to the site. Potable wells within 4 miles of the site, and drawing from the aquifer of concern, serve over 300,000 people.

Division of Environmental Science and Assessment (DESA), Hazardous Waste Support Branch (HWSB), Superfund Contract Support Team (SCST) has been requested by the Environmental Remedial and Response Division (ERRD) to sample the vapors in the on-site building and basements of the residences and businesses in the surrounding vicinity of the site to determine if VOCs in the groundwater may be entering basements/ crawlspaces in vapor form. The on-site building includes a now-former day care facility, Tutor Time, which is where most of the samples will be collected from.

1.2 Project/Task Description:

The purpose of this remedial assessment is to collect valid data which are necessary and efficient to verify that contaminants exist in the residential basements surrounding the site. The sampling event will also determine whether or not an immediate threat to human health or the environment exists. The scope of the removal assessment is to:

- assess the extent of contamination in the on-site building itself, residential basements and businesses; and
- delineate the specific organic contaminants in the vapors.

All analysis of the air samples collected during this sampling event will be performed by the Eastern Research Group, Inc. (ERG). This sampling event will use the ERG contract to supply the SUMMA^{TM} canisters and submit them to their own laboratory for analysis. A copy of the quality assurance project plan for the contract entitled *Support For NMOC/ SNMOC, UATMP, and PAMS Networks, Contract No. 68-D-99-007, Quality Assurance Project Plan* can be found as Appendix B. This document will be referred to as the contractor's or ERG QAPP throughout this document. According to the QAPP, the relevant program for this sampling event is the Urban Air Toxic Monitoring Program (UATMP).

The purpose and scope of this QAPP is to specify the details related to the collection, analysis and validation of the samples collected by the USEPA Region 2, DESA, HWSB, SCST from November 18 - December 10, 2002. The activity schedule is as follows:

ACTIVITY	DATE
Date of the request which initiates the project.	July 11, 2002
Review and Background information	September 05, 2002
Date by which the project plan will be submitted to all interested parties.	October 07, 2002
Obtain site access	Prearranged by ERRD
Date by which comments on the plan are to be received by the project officer.	October 17, 2002
Date(s) of the field reconnaissance.	November 19, 2002
Date(s) of the field sampling activities.	November 18 - December 10, 2002
Date(s) the samples will be submitted to the laboratory for analysis.	All samples will be shipped within 24 hours of collection.
Date(s) by which all analyses are to be completed and the data submitted to the project officer.	30 days.
Date(s) the data will be entered into STORET or other computerized systems.	Not applicable.
Date of the completion of the draft interim/final project report. (Sampling Trip Report)	Within one week of the end of the sampling event
Date by which the reviewer's comments on the report(s) must be received.	Not applicable.
Date for completion of the peer review process.	Not applicable.
Date for the issuance of the final project report.	Within two weeks of receipt of validated analytical data.

The primary use of the data collected will be to determine the extent of air contamination, evaluate potential health risks, and determine environmental impacts. The samples results will be submitted to the Nassau County Health Department to determine whether the contamination is significant enough to cause an adverse effect on human health.

2.0 PROJECT ORGANIZATION AND RESPONSIBILITY

2.1 Project/Task Organization

The following is a list of key personnel and their corresponding responsibilities. Due to the work breakdown structure of the project, an organization list is provided instead of a concise organization chart.

PROJECT PERSONNEL	RESPONSIBILITY
Diane Salkie, Project Officer DESA/HWSB Superfund Contract Support Team	Project Management/ Sampling Operations/ Field Support
Tara Gomes, Environmental Scientist DESA/HWSB Superfund Contract Support Team	Sampling Operations/ Field Support
Michael Mercado, Environmental Scientist DESA/HWSB Superfund Contract Support Team	Sampling Operations/ Field Support
Pat Sheridan, Project Quality Assurance Officer DESA/HWSB/HWSS	Report QA
ERG provided Laboratory	Laboratory Analysis, Laboratory QC, Data Processing Activities, Data Quality Review
Not Applicable	Performance Auditing
Not Applicable	Systems Auditing
DESA/Hazardous Waste Support Branch	Overall QA
Christos Tsiamis, Remedial Project Manager (RPM) ERRD/NYRB	Overall Project Coordination

2.2 Documentation and Records

The data collected for the sampling activities will be organized, analyzed, and summarized in a final project report that will be submitted to the RPM according to the Project Schedule. The report will be prepared by the project officer and include appropriate data quality assessment. Standard methods and references will be used as guidelines for data reduction and reporting. All SOP data generated by the laboratory will be reported in standard deliverable format.

3.0 QUALITY ASSURANCE (QA) OBJECTIVES FOR MEASUREMENT DATA (PARCC)

3.1 Quality Objectives and Criteria for Measurement Data

To assess data quality, PARCC (Precision, Accuracy, Representativeness, Completeness, and Comparability) parameters will be utilized. This is an integral part of the overall monitoring network design. Precision and accuracy are expressed in purely quantitative terms. The other parameters are only expressed using a mixture of quantitative and qualitative terms. All of these parameters are interrelated in terms of overall data quality and they may be difficult to evaluate separately due to these interrelationships. The relative significance of each of the parameters depends on the type and intended use of the data being collected. Therefore, these essential data quality elements are delineated as follows.

3.1.1 Analytical and sample collection precision

The measure of replicate precision is the absolute value of the difference between replicate measurements of the sample divided by the average value and expressed as a percentage as follows:

$$\text{Percent difference} = \frac{|X_1 - X_2|}{X} \times 100$$

where: X_1 - First measurement value
X_2 - Second Measurement value
X - Average of the two values

Factors that affected the precision of the measurement are: molecular weight, water solubility, polarizability, etc. A primary influence is the concentration level of the compound. A replicate precision value of 25 percent can be achieved for each of the target compounds. For more information, refer to Compendium Method *TO-15: Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specialty-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS)* which can be found as Appendix C.

3.1.2 Analytical and sample collection accuracy

A measurement of analytical accuracy is the degree of agreement with audit standards. It is defined as the difference between the nominal concentration of the audit compound and the measured value divided by the audit value and expressed as a percentage as follows:

$$\text{Audit Accuracy, \%} = \frac{\text{Spiked Value - Observed Value}}{\text{Spiked Value}} \times 100$$

For more information, refer to Compendium Method *TO-15: Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specialty-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS)* which can be found as Appendix C. As per Method TO-15, the performance criteria for audit accuracy should be within 30 percent for concentrations normally expected within contaminated ambient air.

3.1.3 Data representativeness

As previously discussed, data representativeness will be assessed by collecting field replicate samples. The field replicates are by definition equally representative of a given point and space and time. Representativeness is a qualitative parameter which is dependent upon the proper design of the sampling program and proper laboratory protocol. Therefore, data representativeness will be satisfied by ensuring that:

The sampling program is followed according to:

> U.S. EPA (Environmental Protection Agency). October 1989. *Region II CERCLA Quality Assurance Manual.* Final Copy, Revision 1. Division of Environmental Services and Assessment, Edison, NJ.; and
>
> U.S. EPA. December 1995. *Superfund Program Representative Sampling Guidance.* OSWER Directive 9360.4-10. Interim Final. EPA/540/R-95/141. Office of Emergency and Remedial Response (OERR). Washington, D.C.

Proper sampling techniques are used in accordance with:

> U.S. EPA. *Environmental Response Team (ERT) Standard Operating Procedure (SOP) #1704: Summa Canister Sampling;* revised July 1995. The SOP is enclosed in Appendix D.
>
> Eastern Research Group (ERG). March 2000. + *Support For NMOC/ SNMOC, UATMP, and PAMS Networks, Contract No. 68-D-99-007, Quality Assurance Project Plan*. Morrisville, NC. This QAPP is enclosed as Appendix B.

Proper analytical procedures are followed and holding times of the samples are not exceeded in the laboratory according to:

> U.S. EPA. January 1999. Compendium Method *TO-15: Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specialty-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS)* from the *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air.* Second Edition. Center for Environmental Research Information. Office of Research and Development. Cincinnati, OH which can be found as Appendix C.

3.1.4 Data completeness

Data completeness will be expressed as the percentage of valid data obtained from measurement system. For data to be considered valid, it must meet all the acceptable criteria including accuracy and precision, as well as any other criteria specified by the analytical method used. Therefore, all data points critical to the sampling program in terms of completeness will be 100% validated by ERG according to Section 15 of ERG's QAPP which can be found as Appendix B. With 100% validation, the rationale for considering data points non-critical is not required.

3.1.5 Data comparability

To ensure data comparability, sampling and analysis for all samples will be performed using standardized analytical methods and adherence to the quality control procedures outlined in the methods and this QAPP. Therefore, the data will be comparable.

4.0 SAMPLING PROCEDURES

4.1 Sampling Process Design

As part of the remedial assessment process, U.S. EPA Region II DESA/HWSB/SCST personnel will collect air samples from the on-site building and in the basements/ crawlspaces of houses and businesses in the area of the Jackson Steel site. Samples will be collected with SUMMA™ canisters. SUMMA™ Canister sampling will follow methods as described in U.S. EPA/ERT SOP #1704, Appendix D.

The sampling design for the site, including the rationale for sample frequency, location, and depth was predetermined by the remedial project manager (RPM). For the purposes of this sampling event, sample location selection was determined by selecting locations of in the area suspected ground water contamination. Each location to be sampled will be chosen by the RPM prior to sampling. The resident/owner will be notified prior to the sampling day by a telephone call and an instruction page through the mail. A copy of this page can be found as Appendix H. The resident/owner will also be required to sign an access agreement, allowing the EPA to enter their home and sample the air in their basement. An example of this can be found as Appendix G. A map of the area can be found in Appendix A. A detailed description of sample collection methodology is presented in Section 4, Sub-section 2, Part 2: Sample Collection Methodology.

The following locations will be sampled as follows: ten (10) from Tutor Time with ventillation system on, one (1) from the billiard hall with ventillation system on, four (4) from residents on First Street, five (5) from apartment building east of the site and three (3) from outside of the vents of the subvent system in the main on-site building. Two (2) samples will be collected from the ambient outside air and up to four (4) samples will be duplicate samples collected from the same location. Three weeks later, ten (10) more samples will be collected from Tutor Time with the ventillation system off and one (1) more sample from the billiard hall with the ventillation system off. All samples will be analyzed for *Title III Clean Air Amendment List* - volatile organic compounds (VOC) which can be found in Appendix F. All samples will be collected by U.S. EPA personnel and then sent to ERG contractors. The contractors are providing the canisters and submitting the samples to their own laboratory who will analyze the samples according to *TO-15* which can be found as Appendix C.

The sampling and analysis protocol is listed as Table 1 on page 8.

TABLE 1
Jackson Steel Site
Remedial Investigation
Sampling and Analysis Protocols

Sample Type	Number of Samples	Matrix	Parameter/Fraction	Sample Container	Sample Preservation	Analytical Method [1]	Method Detection Limit	Holding Time
On-site Building/ Residential Basements	40	Air	Title III Clean Air Amendment List Volatile Organic Compounds (VOCs)	(1) SUMMA Canister	---------	*TO-15*	0.04 - 0.26 ppbv	30 days

Legend:

[1] U.S. EPA. January 1999. Compendium Method *TO-15: Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specialty-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS)* from the *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air*. Second Edition.

4.2 Sampling Methods Requirements

4.2.1 Standard operating procedures

As previously stated, all sampling will be in accordance with the *U.S. EPA Region II CERCLA Quality Assurance Manual*; and *U.S. EPA Superfund Program Representative Sampling Guidance* OSWER Directive 9360.4-10, Interim Final, EPA/540/R-95/141, Office of Emergency and Remedial Response (OERR), Washington, D.C. Furthermore, the specific Standard Operating Procedure (SOP) utilized for air sampling, as presented in Appendix D, is the *U.S. EPA ERT SOP #1704: Summa Canister Sampling*.

4.2.2 Sample collection methodology

All samples including QA/QC samples will be collected by personnel from the USEPA Region II DESA/HWSB/SCST from the on-site building and residential and business basements/crawlspaces in the area of the Jackson Steel site. The total number of samples includes: thirty four (34) field samples in addition to: four (4) laboratory quality control samples (i.e. field duplicate) and two (2) ambient air sample. Samples will be collected by placing a SUMMA™ canister in the residential basement, connecting the pre-set valve for the appropriate amount of time and retrieving the canister after 24 hours. The sampler will also ask the resident/owner questions from the *Questionnaire* and survey the chemicals found in the basement/crawlspace.

4.2.3 Sample Containers, Volume, Preservation, and Holding Times

Sample container type, volume, preservation, and holding times are dependent upon analytical parameter and fraction and are matrix specific. The following table outlines the sample container type, volume, preservation, and holding times for samples to be collected on-site.

Analytical Parameter/Fraction	Sample Container	Required Sample Volume	Sample Preservation	Holding Time
Clean Air Amendment List - VOC	(1) SUMMA™ canister	6 lts.	---------	30 days to analyze

4.2.4 Field measurement data collection

Canister Sample Data Sheets, *Questionnaires*, *Chain of Custody* and the field notebook will be completed for each sample collected. The *Questionnaire* will record sample location; residential information; time of sample drop off and pick up; conditions in the room; chemicals stored in the room; laboratory sample number; laboratory sample analysis and sample collection notes and/or observations. An example of the *Questionnaire* is presented in Appendix G. The *Canister Sample Data Sheet* will be provided by ERG and records the sample location, sampling period, initial and final sample time and comments. An example of this data sheet can also be found in Appendix G. The *Chain of Custody* is a record of the sample location, sample canister and valve numbers and time and date of the sample. An example of the *Chain of Custody* can be found in Appendix G. The field notebook will be completed as provided for in Section 8.4: Data Quality Management of the QAPP.

4.2.5 Sampling Equipment Decontamination

Air samples will be collected using SUMMA™ canisters. ERG will perform decontamination of the canister prior to sending them to U.S. EPA. The SUMMA™ canisters will be cleaned according to:

- *Support For NMOC/ SNMOC, UATMP, and PAMS Networks*, *Contract No. 68-D-99-007, Quality Assurance Project Plan* (Appendix B);
- *U.S. EPA ERT SOP #1703: Sample Documentation* (Appendix E); and
- *U.S. EPA Region II CERCLA Quality Assurance Manual.*

4.2.6 Management of Investigative-Derived Wastes (IDW)

The wastes that are anticipated on being generated during this sampling event are personnel protective equipment (i.e. goggles, booties, etc.). The personnel protective equipment will be double-bagged and properly disposed of in on-site solid waste roll-off or off-site in properly designated containers.

5.0 SAMPLE CUSTODY

5.1 Special Training Requirements/Certification

To perform the operations of this sampling event, SCST will be dealing with the remedial activities on-site. This can imminently expose SCST personnel to potential occupational environmental hazards. As a result, it is important for SCST field personnel to be familiar with:

- Identifying methods and procedures for recognizing, evaluating and controlling hazardous substances.
- Identifying concepts, principles, and guidelines to properly protect SCST field personnel.
- Discussing regulations and action levels to ensure the health and safety of SCST field oversight personnel.
- Discussing the fundamentals needed to develop organizational structures and standard operating procedures to mitigate potential environmental hazards.
- Demonstrating the selection and use of dermal and respiratory protective equipment.
- Demonstrating the selection and use of direct-reading air monitoring instrumentation (if applicable).

In practice, not all of the potential environmental hazards which may be inherent to a site can be readily anticipated. To mitigate these circumstances, SCST field personnel must learn, follow, and enforce the published rules governing occupational health and safety. In addition, they must maintain awareness and exercise common sense and good judgement when confronting possible unsafe situations. Consequently, all divisions and offices at the Edison facility are required to provide their staff with the necessary safety training and equipment to perform their assigned duties.

For SCST personnel, all training and certification requirements are to be undertaken in accordance with the protocols set forth in the 1995 "Edison Health and Safety Manual." Specifically, this requires completion of the forty (40) hour "Hazardous Materials Incident Response Operations" training pursuant to Occupational Safety and Health Administration (OSHA) regulation 29 CFR 1910.120 and U.S. EPA Order 1440.2. This is to be supplemented by completing the twenty four (24) hour OSHA sanctioned supervised on-site operations certification training. In conjunction, SCST personnel are also to maintain certifications for:

- The supplemental eight (8) hour annual health and safety refresher training.
- Fit testing for atmosphere supplying respirators (Level B) and air purifying respirators (Level C).
- Enrollment in a physician authorized medical monitoring program.

5.2 Sample Handling and Custody Requirements

5.2.1 Sample handling and shipment

Canister Sample Data Sheets, a *Questionnaire, Chain of Custody* and the field notebook will be completed for each sample collected. All field and sample documents will be legibly written in indelible ink. Any corrections or revisions will be made by lining through the original entry and initialing the change. The *Questionnaire* will record sample location; residential information; time of sample drop off and pick up; conditions in the room; laboratory sample number; laboratory sample analysis and sample collection notes and/or observations. For reference, an example of the *Questionnaire* is presented as Appendix G. The *Canister Sample Data Sheet* will be provided by ERG and records the sample location, sampling period, initial and final sample time and comments. An example of this data sheet can also be found in Appendix G. The *Chain of Custody* is a record of the sample location, sample canister and valve numbers and time and date of the sample. An example of the *Chain of Custody* can be found in Appendix G. The field notebook will be used by field personnel to record all aspects of sample collection and handling, visual observations, and field measurements. The field notebook is a descriptive notebook detailing site activities and observations so that an accurate, factual account of field procedures may be reconstructed. The sample team or individuals performing a particular sampling activity are required to maintain a field notebook. This field notebook will be a bound weatherproof logbook that shall be filled out at the location of sample collection immediately after sampling. All entries will be signed by the individuals making them. At a minimum, the notebook will contain sample particulars including sample number, collection time, location, descriptions, methods used, daily weather conditions, field measurements, name of sampler(s), sample preservation, names of contractor/ subcontractor personnel, and other site-specific observations including any deviations from protocol.

The *Canister Tag*, found in Appendix G, also provided by ERG, will be securely affixed to each SUMMA^{TM} canister and include only the sample identification number as per protocol. The sample tags will be secured to the canister itself. Once sealed, samples will be placed back into the cardboard boxes that they were received in. Custody seals and strapping tape will then be affixed to the boxes.

Samples will be packaged and shipped in accordance with USEPA, Department of Transportation (DOT), and International Air Transport Association (IATA) procedures. All samples will be shipped within 24 hours of collection to the ERG office in North Carolina.

5.2.2 Sample custody procedures

Standard U.S.EPA Chain-of-Custody Procedures will be followed for all samples and be in accordance with the U.S.EPA Region II *CERCLA Quality Assurance Manual*. The *Chain of Custody* records will be maintained from the time of sample collection until final deposition. Every transfer of custody will be noted and signed for and a copy of the record will be kept for each individual who has signed it. The *Chain of Custody* records will include, at a minimum, sample identification number, number of samples collected, sample collection date and time, sample type, sample matrix, sample container type, sample analysis requested, sample preservation, and the name(s) and signature(s) of samplers and all individuals who have had custody. Sample labels will only include the sample identification number as per protocol to prevent any conflict of interest issues. Custody seals will demonstrate that a sample container or cooler has not been opened or tampered with. The sampler will sign and date the custody seal and affix it to the container and/or cooler in such a manner that it cannot be opened without breaking the seal.

8.4 Data Quality Management

All project data and information must be documented in a format that is usable by project personnel. This section of the QAPP describes how project data and information will be documented, tracked, and managed from their generation in the field to final use and storage in a manner that ensures data integrity and defensibility. All field and sample documents will be legibly written in indelible ink. Any correction or revisions will be made by lining through the original entry and initialing the change.

The following field and sample documentation will be maintained.

- The field notebook is a descriptive notebook detailing site activities and observations so that an accurate, factual account of field procedures may be reconstructed. The sample team or individuals performing a particular sampling activity are required to maintain a field notebook. This field notebook will be a bound weatherproof logbook that shall be filled out at the location of sample collection immediately after sampling. All entries will be signed by the individuals making them. At a minimum, the notebook will contain sample particulars including sample number, collection time, location, descriptions, methods used, daily weather conditions, field measurements, name of sampler(s), sample preservation, and other site-specific observations including any deviations from protocol.

- Field data sheets, i.e., *Questionnaire, Canister Field Data Sheet*, and corresponding sample labels are used to identify samples and document field sampling conditions and activities. The field data sheets will be completed at the time of sample collection and will include the following: sample location; residential information; drop off and pick up time; sample environment description; laboratory sample number; laboratory sample analysis; and sample collection notes and/or observations. An example of the *Questionnaire* and the *Canister Field Data Sheet* are presented in Appendix G. Sample labels will be securely affixed to the sample container and include only the sample identification number as per Protocol.

- Sample tags will be securely affixed to the sample container and include only the sample identification number as per protocol to prevent any conflict of interest issues. The sample labels will be sealed to a bag surrounding the canister sample label integrity.

- The *Chain of Custody* records will be maintained from the time of sample collection until final deposition. Every transfer of custody will be noted and signed for and a copy of the record will be kept for each individual who has signed it. The chain-of-custody records will include, at a minimum, sample identification number, number of samples collected, sample collection date and time, sample type, sample matrix, sample container type, sample analysis requested, sample preservation, and the name(s) and signature(s) of samplers and all individuals who have had custody. An example of the chain of custody that will be used at this site can be found in Appendix G.

6.0 CALIBRATION PROCEDURES AND FREQUENCY

6.1 Instrument Calibration and Frequency

Laboratory analytical equipment calibration will follow procedures as specified under U.S. EPA, Compendium Method *TO-15: Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specialty-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS)* from the *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air*, which can be found as Appendix C.

7.0 ANALYTICAL PROCEDURES

7.1 Analytical Methods Requirements

The analytical method, equipment and method performance requirements for analysis will be according to ERG's contract. Refer to U.S. EPA, Compendium Method *TO-15: Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specialty-Prepared Canisters and Analyzed by Gas Chromatography/ Mass Spectrometry (GC/MS)* from the *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air*, which can be found as Appendix C.

8.0 DATA REDUCTION, VALIDATION, AND REPORTING

8.1 Data Review, Validation and Verification Requirements:

Standard methods and references will be used as guidelines for data reduction and reporting. All data generated by the laboratory will be reported in standard deliverable format. ERG will be using a gas chromatograph (GC)/flame ionization detector (FID)/ mass selective detector (MSD) to analyze the samples for VOCs as stated in their QAPP which is based on TO-15. Due to ERG's vast experience with analyzing SUMMA^{TM} canisters for VOCs, they have found this method to be the most precise and they are able to detect the compounds at unusually low concentrations. All data validation reports will be summarized according ERG's QAPP: *Support For NMOC/ SNMOC, UATMP, and PAMS Networks, Contract No. 68-D-99-007, Quality Assurance Project Plan, Section 15* which can be found as Appendix B.

8.2 Validation and Verification Methods

All data will be validated by ERG's generic *Support For NMOC/ SNMOC, UATMP, and PAMS Networks, Contract No. 68-D-99-007, Quality Assurance Project Plan, Section 15,* which can be found as Appendix B.

8.3 Data Acquisition Requirements

Data acquisition from non-direct measurements such as data from databases or literature is not anticipated at this time. Therefore, this is not applicable.

- Custody seals will demonstrate that a sample canister or box has not been opened or tampered with. The sampler will sign and date the custody seal and affix it to the box in such a manner that it cannot be opened without breaking the seal.

- Procedures are provided for project personnel to make changes, take corrective actions and document the process through Corrective Action Request Forms. Corrective action can occur during field activities, laboratory analysis, data validation, and data assessment. For further information, refer to Section 13.0: Corrective Action.

9.0 INTERNAL QUALITY CONTROL CHECKS AND FREQUENCY

9.1 Quality Control Requirements

As previously stated, to assess data quality, PARCC (Precision, Accuracy, Representativeness, Completeness, and Comparability) parameters will be utilized. These essential data quality elements are delineated as follows.

9.1.1 Data precision

Precision is defined as a measure of the reproducibility of individual measurements of the same property under a given set of conditions. The overall precision of measurement data is a mixture of sampling and analytical factors.

9.1.1.1 Analytical precision

The measure of replicate precision is the absolute value of the difference between replicate measurements of the sample divided by the average value and expressed as a percentage as follows:

$$\text{Percent difference} = \frac{|X_1 - X_2|}{X} \times 100$$

where: X_1 - First measurement value
X_2 - Second Measurement value
X - Average of the two values

Factors that affected the precision of the measurement are: molecular weight, water solubility, polarizability, etc. A primary influence is the concentration level of the compound. A replicate precision value of 25 percent can be achieved for each of the target compounds. For more information, refer to Compendium Method *TO-15: Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specialty-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS)* which can be found as Appendix C.

Table 2 located on page 17 of this QAPP depicts the analytical precision for the analytical methods chosen in terms of estimated relative percent difference (RPD).

9.1.1.2 Sample collection precision

Sample collection precision will be assessed by collecting field replicate samples. The field replicates will be used to evaluate errors associated with sample heterogeneity, sampling methodology and analytical procedures. The analytical results from these samples will provide data on the overall measurement precision.

9.1.2 Data accuracy

Accuracy is defined as the degree of difference between measured or calculated values and the true value. The closer the numerical value of the measurement comes to the true value, or actual concentration, the more accurate the measurement is. It is difficult to measure accuracy for the entire data collection activity. Sources of error are the sampling process, field contamination, preservation, handling, sample matrix, sample preparation and analysis techniques.

9.1.2.1 Analytical accuracy

A measurement of analytical accuracy is the degree of agreement with audit standards. It is defined as the difference between the nominal concentration of the audit compound and the measured value divided by the audit value and expressed as a percentage as follows:

$$\text{Audit Accuracy, \%} = \frac{\text{Spiked Value - Observed Value}}{\text{Spiked Value}} \text{ X } 100$$

For more information, refer to Compendium Method *TO-15: Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specialty-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS)* which can be found as Appendix C.

Table 2 on page 17 located in this QAPP depicts both the analytical precision and accuracy for the analytical methods chosen in terms of estimated percent recovery.

9.1.2.2 Sample collection accuracy

Method blanks will be used to monitor possible laboratory contamination.

9.1.3 Data Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, or and environmental condition. Representativeness is a qualitative parameter which is most concerned with the proper design of the sampling program and proper laboratory protocol. The representativeness criterion is best satisfied by making certain that sampling locations are selected properly and a sufficient number of samples are collected. Therefore, data representativeness will be assessed by collecting field replicate samples. The field replicates are by definition equally representative of a given point in space and time.

In addition, as previously stated, data representativeness will be satisfied by ensuring that the sampling program is followed according to the *U.S. EPA Region II CERCLA Quality Assurance Manual*; and the *U.S. EPA Superfund Program Representative Sampling Guidance* for soil, Volume 1. Also, proper sampling techniques will be used in accordance with the U.S. EPA. *Environmental Response Team (ERT) Standard Operating Procedure (SOP) #1704: Summa Canister Sampling.* The SOP is enclosed in Appendix D.

9.1.4 Data Comparability

Comparability is defined as the confidence with which one data set can be compared to another. Field and laboratory procedures greatly affect comparability. Therefore, to optimize comparability, sampling and analysis for all samples will be performed using standardized analytical methods and adherence to the quality control procedures outlined in the methods and this QAPP. Therefore, the data will be compared.

9.1.5 Data Completeness

Completeness is defined as the measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. Data completeness will be expressed as the percentage of valid data obtained from measurement system. For data to be considered valid, it must meet all the acceptable criteria including accuracy and precision, as well as any other criteria specified by the analytical method used. Therefore, all data points critical to the sampling program in terms of completeness will be 100% validated by the ERG contract in accordance with the ERG's generic *Support For NMOC/ SNMOC, UATMP, and PAMS Networks, Contract No. 68-D-99-007, Quality Assurance Project Plan, Section 15.* With 100% validation, the rationale for considering data points non-critical is not required.

10.0 Performance and Systems Audits

10.1 Assessments and Response Actions

No performance audit of field operations is anticipated at this time. If conducted, performance and systems audits will be in accordance with:

- U.S. EPA (Environmental Protection Agency) Region II. April 2000. *SOP SCST-1, Standard Operating Procedure (S.O.P.) for Performing Oversight of CERCLA Field Operations.* Revision 0. Division of Environmental Services and Assessment, Hazardous Waste Support Branch, Hazardous Waste Support Section, Edison, NJ.

11.0 PREVENTIVE MAINTENANCE

11.1 Instrument/Equipment Testing, Procedures & Scheduled Inspection and Maintenance Requirements

As previously stated, calibration and preventative maintenance of analytical laboratory equipment will follow procedures as specified in ERG's generic *Support For NMOC/ SNMOC, UATMP, and PAMS Networks, Contract No. 68-D-99-007, Quality Assurance Project Plan, Section 15,* which can be found as Appendix B.

11.2 Inspection/Acceptance Requirements for Supplies and Consumables

Due to the nature of air sampling rinsate and trip blanks are not applicable. SUMMA™ canister quality control includes calibration of the canister itself, method blanks performed by the laboratory and laboratory control samples, also performed by the laboratory.

12.0 SPECIFIC ROUTINE PROCEDURES/MEASUREMENT PARAMETERS INVOLVED

12.1 Reconciliation with Data Used to Assess PARCC for Quality Objectives Measurement

Sample collection precision will be evaluated by collecting and analyzing a field duplicate sample. The field duplicate samples will be used to evaluate errors associated with sample heterogeneity, sampling methodology and analytical procedures. The analytical results from the field duplicate will provide data on the overall measurement precision. Precision will be reported as the relative percent difference (RPD) for two measurements. The acceptance criteria for the field duplicate samples are located in Table 2 on page 17.

Data will be generated through the collection of air samples in the on-site building, residential and business basements/crawlspaces in the area of the Jackson Steel site. This data will be used to determine the location and concentration of contamination in the residents, the extent of contamination, evaluate potential health threats, and determine environmental impacts while identifying clean-up criteria.

TABLE 2: PRECISION AND ACCURACY

Sample Parameter/Fraction	Sample Matrix	Analytical Method	Method Detection Limit[1]	Quantitation Limit	Estimated Accuracy[2]	Accuracy Protocol	Estimated Precision[2]	Precision Protocol
Title III Clean Air Amendment List Volatile Organic Compounds (VOCs)	Air	*TO-15*	0.04 -0.26 ppbv	ppbv	< or = 30%	Non-RAS	25%	Non-RAS

[1] The method detection limits were provided by ERG as per their generic *Support For NMOC/ SNMOC, UATMP, and PAMS Networks, Contract No. 68-D-99-007, Quality Assurance Project Plan.*

[2] *TO-15: Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specialty-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS)*

13.0 CORRECTIVE ACTION

13.1 Assessments and Response Actions

Procedures are provided for project personnel to make changes, take corrective actions and document the process through Corrective Action Request Forms. Corrective action can occur during field activities, laboratory analysis, data validation, and data assessment.

Corrective action in the field may be necessary when the monitoring network design is changed. A change in the field includes: increasing the number or type of samples or analyses; changing sampling locations; and/or modifying sampling protocol. When this occurs, the project officer or project QA officer will identify any suspected technical or QA deficiencies and note them in the field logbook. The project QA officer will be responsible for assessing the suspected deficiency and determining the impact on the quality of the data. Development of the appropriate corrective action will be the responsibility of the RPM.

Data validation and data assessment corrective action will be in accordance with the *U.S. EPA Region II CERCLA Quality Assurance Manual.*

14.0 QA REPORTS TO MANAGEMENT

14.1 Distribution List

The following project personnel will receive copies of the approved QAPP and any subsequent revisions.

Project Personnel	Title
Christos Tsiamis ERRD/NYRB	Remedial Project Manager
Diane Salkie DESA/HWSB	Project Officer
Pat Sheridan DESA/HWSB	Quality Assurance Officer

14.2 Reports to Management

The data collected as a result of sampling activities; will be organized, analyzed and summarized in a final project report that will be submitted to the RPM according to the Project Schedule. The report will be prepared by the project officer or project quality assurance officer and include appropriate data quality assessment.

APPENDIX A

SITE MAPS

APPENDIX C

U.S. Environmental Protection Agency (EPA)

Compendium Method *TO-15:*
Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specialty-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS)
from the
Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air
Second Edition

Center for Environmental Research Information

Office of Research and Development. Cincinnati, OH
January 1999

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

January 1999

Method TO-15
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DISCLAIMER

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

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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)

1. Scope

1.1 This method documents sampling and analytical procedures for the measurement of subsets of the 97 volatile organic compounds (VOCs) that are included in the 189 hazardous air pollutants (HAPs) listed in Title III of the Clean Air Act Amendments of 1990. VOCs are defined here as organic compounds having a vapor pressure greater than 10^{-1} Torr at 25°C and 760 mm Hg. Table 1 is the list of the target VOCs along with their CAS number, boiling point, vapor pressure and an indication of their membership in both the list of VOCs covered by Compendium Method TO-14A (1) and the list of VOCs in EPA's Contract Laboratory Program (CLP) document entitled: *Statement-of-Work (SOW) for the Analysis of Air Toxics from Superfund Sites (2).*

Many of these compounds have been tested for stability in concentration when stored in specially-prepared canisters (see Section 8) under conditions typical of those encountered in routine ambient air analysis. The stability of these compounds under all possible conditions is not known. However, a model to predict compound losses due to physical adsorption of VOCs on canister walls and to dissolution of VOCs in water condensed in the canisters has been developed (3). Losses due to physical adsorption require only the establishment of equilibrium between the condensed and gas phases and are generally considered short term losses. (i.e., losses occurring over minutes to hours). Losses due to chemical reactions of the VOCs with cocollected ozone or other gas phase species also account for some short term losses. Chemical reactions between VOCs and substances inside the canister are generally assumed to cause the gradual decrease of concentration over time (i.e., long term losses over days to weeks). Loss mechanisms such as aqueous hydrolysis and biological degradation (4) also exist. No models are currently known to be available to estimate and characterize all these potential losses, although a number of experimental observations are referenced in Section 8. Some of the VOCs listed in Title III have short atmospheric lifetimes and may not be present except near sources.

1.2 This method applies to ambient concentrations of VOCs above 0.5 ppbv and typically requires VOC enrichment by concentrating up to one liter of a sample volume. The VOC concentration range for ambient air in many cases includes the concentration at which continuous exposure over a lifetime is estimated to constitute a 10^{-6} or higher lifetime risk of developing cancer in humans. Under circumstances in which many hazardous VOCs are present at 10^{-6} risk concentrations, the total risk may be significantly greater.

1.3 This method applies under most conditions encountered in sampling of ambient air into canisters. However, the composition of a gas mixture in a canister, under unique or unusual conditions, will change so that the sample is known not to be a true representation of the ambient air from which it was taken. For example, low humidity conditions in the sample may lead to losses of certain VOCs on the canister walls, losses that would not happen if the humidity were higher. If the canister is pressurized, then condensation of water from high humidity samples may cause fractional losses of water-soluble compounds. Since the canister surface area is limited, all gases are in competition for the available active sites. Hence an absolute storage stability cannot be assigned to a specific gas. Fortunately, under conditions of normal usage for sampling ambient air, most VOCs can be recovered from canisters near their original concentrations after storage times of up to thirty days (see Section 8).

1.4 Use of the Compendium Method TO-15 for many of the VOCs listed in Table 1 is likely to present two difficulties: (1) what calibration standard to use for establishing a basis for testing and quantitation, and (2) how to obtain an audit standard. In certain cases a chemical similarity exists between a thoroughly tested compound and others on the Title III list. In this case, what works for one is likely to work for the other in terms of making standards. However, this is not always the case and some compound standards will be troublesome. The reader is referred to the Section 9.2 on standards for guidance. Calibration of compounds such as formaldehyde, diazomethane, and many of the others represents a challenge.

1.5 Compendium Method TO-15 should be considered for use when a subset of the 97 Title III VOCs constitute the target list. Typical situations involve ambient air testing associated with the permitting procedures for emission sources. In this case sampling and analysis of VOCs is performed to determine the impact of dispersing source emissions in the surrounding areas. Other important applications are prevalence and trend monitoring for hazardous VOCs in urban areas and risk assessments downwind of industrialized or source-impacted areas.

1.6 Solid adsorbents can be used in lieu of canisters for sampling of VOCs, provided the solid adsorbent packings, usually multisorbent packings in metal or glass tubes, can meet the performance criteria specified in Compendium Method TO-17 which specifically addresses the use of multisorbent packings. The two sample collection techniques are different but become the same upon movement of the sample from the collection medium (canister or multisorbent tubes) onto the sample concentrator. Sample collection directly from the atmosphere by automated gas chromatographs can be used in lieu of collection in canisters or on solid adsorbents.

2. Summary of Method

2.1 The atmosphere is sampled by introduction of air into a specially-prepared stainless steel canister. Both subatmospheric pressure and pressurized sampling modes use an initially evacuated canister. A pump ventilated sampling line is used during sample collection with most commercially available samplers. Pressurized sampling requires an additional pump to provide positive pressure to the sample canister. A sample of air is drawn through a sampling train comprised of components that regulate the rate and duration of sampling into the pre-evacuated and passivated canister.

2.2 After the air sample is collected, the canister valve is closed, an identification tag is attached to the canister, and the canister is transported to the laboratory for analysis.

2.3 Upon receipt at the laboratory, the canister tag data is recorded and the canister is stored until analysis. Storage times of up to thirty days have been demonstrated for many of the VOCs (5).

2.4 To analyze the sample, a known volume of sample is directed from the canister through a solid multisorbent concentrator. A portion of the water vapor in the sample breaks through the concentrator during sampling, to a degree depending on the multisorbent composition, duration of sampling, and other factors. Water content of the sample can be further reduced by dry purging the concentrator with helium while retaining target compounds. After the concentration and drying steps are completed, the VOCs are thermally desorbed, entrained in a carrier gas stream, and then focused in a small volume by trapping on a reduced temperature trap or small volume multisorbent trap. The sample is then released by thermal desorption and carried onto a gas chromatographic column for separation.

As a simple alternative to the multisorbent/dry purge water management technique, the amount of water vapor in the sample can be reduced below any threshold for affecting the proper operation of the analytical system by reducing the sample size. For example, a small sample can be concentrated on a cold trap and released directly to the gas chromatographic column. The reduction in sample volume may require an enhancement of detector sensitivity.

Other water management approaches are also acceptable as long as their use does not compromise the attainment of the performance criteria listed in Section 11. A listing of some commercial water management systems is provided in Appendix A. One of the alternative ways to dry the sample is to separate VOCs from condensate on a low temperature trap by heating and purging the trap.

2.5 The analytical strategy for Compendium Method TO-15 involves using a high resolution gas chromatograph (GC) coupled to a mass spectrometer. If the mass spectrometer is a linear quadrupole system, it is operated either by continuously scanning a wide range of mass to charge ratios (SCAN mode) or by monitoring select ion monitoring mode (SIM) of compounds on the target list. If the mass spectrometer is based on a standard ion trap design, only a scanning mode is used (note however, that the Selected Ion Storage (SIS) mode for the ion trap has features of the SIM mode). Mass spectra for individual peaks in the total ion chromatogram are examined with respect to the fragmentation pattern of ions corresponding to various VOCs including the intensity of primary and secondary ions. The fragmentation pattern is compared with stored spectra taken under similar conditions, in order to identify the compound. For any given compound, the intensity of the primary fragment is compared with the system response to the primary fragment for known amounts of the compound. This establishes the compound concentration that exists in the sample.

Mass spectrometry is considered a more definitive identification technique than single specific detectors such as flame ionization detector (FID), electron capture detector (ECD), photoionization detector (PID), or a multidetector arrangement of these (see discussion in Compendium Method TO-14A). The use of both gas chromatographic retention time and the generally unique mass fragmentation patterns reduce the chances for misidentification. If the technique is supported by a comprehensive mass spectral database and a knowledgeable operator, then the correct identification and quantification of VOCs is further enhanced.

3. Significance

3.1 Compendium Method TO-15 is significant in that it extends the Compendium Method TO-14A description for using canister-based sampling and gas chromatographic analysis in the following ways:

- Compendium Method TO-15 incorporates a multisorbent/dry purge technique or equivalent (see Appendix A) for water management thereby addressing a more extensive set of compounds (the VOCs mentioned in Title III of the CAAA of 1990) than addressed by Compendium Method TO-14A. Compendium Method TO-14A approach to water management alters the structure or reduces the sample stream concentration of some VOCs, especially water-soluble VOCs.
- Compendium Method TO-15 uses the GC/MS technique as the only means to identify and quantitate target compounds. The GC/MS approach provides a more scientifically-defensible detection scheme which is generally more desirable than the use of single or even multiple specific detectors.
- In addition, Compendium Method TO-15 establishes method performance criteria for acceptance of data, allowing the use of alternate but equivalent sampling and analytical equipment. There are several new and viable commercial approaches for water management as noted in Appendix A of this method on which to base a VOC monitoring technique as well as other approaches to sampling (i.e., autoGCs and solid

adsorbents) that are often used. This method lists performance criteria that these alternatives must meet to be acceptable alternatives for monitoring ambient VOCs.

- Finally, Compendium Method TO-15 includes enhanced provisions for inherent quality control. The method uses internal analytical standards and frequent verification of analytical system performance to assure control of the analytical system. This more formal and better documented approach to quality control guarantees a higher percentage of good data.

3.2 With these features, Compendium Method TO-15 is a more general yet better defined method for VOCs than Compendium Method TO-14A. As such, the method can be applied with a higher confidence to reduce the uncertainty in risk assessments in environments where the hazardous volatile gases listed in the Title III of the Clean Air Act Amendments of 1990 are being monitored. An emphasis on risk assessments for human health and effects on the ecology is a current goal for the U.S. EPA.

4. Applicable Documents

4.1 ASTM Standards

- **Method D1356** *Definitions of Terms Relating to Atmospheric Sampling and Analysis.*
- **Method E260** *Recommended Practice for General Gas Chromatography Procedures.*
- **Method E355** *Practice for Gas Chromatography Terms and Relationships.*
- **Method D5466** *Standard Test Method of Determination of Volatile Organic Compounds in Atmospheres (Canister Sampling Methodology).*

4.2 EPA Documents

- *Quality Assurance Handbook for Air Pollution Measurement Systems. Volume II,* U. S. Environmental Protection Agency, EPA-600/R-94-038b, May 1994.
- *Technical Assistance Document for Sampling and Analysis of Toxic Organic Compounds in Ambient Air.* U. S. Environmental Protection Agency, EPA-600/4-83-027, June 1983.
- *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air: Method TO-14, Second Supplement,* U. S. Environmental Protection Agency, EPA-600/4-89-018, March 1989.
- *Statement-of-Work (SOW) for the Analysis of Air Toxics from Superfund Sites,* U. S. Environmental Protection Agency, Office of Solid Waste, Washington, D.C., Draft Report, June 1990.
- *Clean Air Act Amendments of 1990,* U. S. Congress, Washington, D.C., November 1990.

5. Definitions

[Note: Definitions used in this document and any user-prepared standard operating procedures (SOPs) should be consistent with ASTM Methods D1356, E260, and E355. Aside from the definitions given below, all pertinent abbreviations and symbols are defined within this document at point of use.]

5.1 Gauge Pressure—pressure measured with reference to the surrounding atmospheric pressure, usually expressed in units of kPa or psi. Zero gauge pressure is equal to atmospheric (barometric) pressure.

5.2 **Absolute Pressure**—pressure measured with reference to absolute zero pressure, usually expressed in units of kPa, or psi.

5.3 **Cryogen**—a refrigerant used to obtain sub-ambient temperatures in the VOC concentrator and/or on front of the analytical column. Typical cryogens are liquid nitrogen (bp -195.8°C), liquid argon (bp -185.7°C), and liquid CO_2 (bp -79.5°C).

5.4 **Dynamic Calibration**—calibration of an analytical system using calibration gas standard concentrations in a form identical or very similar to the samples to be analyzed and by introducing such standards into the inlet of the sampling or analytical system from a manifold through which the gas standards are flowing.

5.5 **Dynamic Dilution**—means of preparing calibration mixtures in which standard gas(es) from pressurized cylinders are continuously blended with humidified zero air in a manifold so that a flowing stream of calibration mixture is available at the inlet of the analytical system.

5.6 **MS-SCAN**—mass spectrometric mode of operation in which the gas chromatograph (GC) is coupled to a mass spectrometer (MS) programmed to SCAN all ions repeatedly over a specified mass range.

5.7 **MS-SIM**—mass spectrometric mode of operation in which the GC is coupled to a MS that is programmed to scan a selected number of ions repeatedly [i.e., selected ion monitoring (SIM) mode].

5.8 **Qualitative Accuracy**—the degree of measurement accuracy required to correctly identify compounds with an analytical system.

5.9 **Quantitative Accuracy**—the degree of measurement accuracy required to correctly measure the concentration of an identified compound with an analytical system with known uncertainty.

5.10 **Replicate Precision**—precision determined from two canisters filled from the same air mass over the same time period and determined as the absolute value of the difference between the analyses of canisters divided by their average value and expressed as a percentage (see Section 11 for performance criteria for replicate precision).

5.11 **Duplicate Precision**—precision determined from the analysis of two samples taken from the same canister. The duplicate precision is determined as the absolute value of the difference between the canister analyses divided by their average value and expressed as a percentage.

5.12 **Audit Accuracy**—the difference between the analysis of a sample provided in an audit canister and the nominal value as determined by the audit authority, divided by the audit value and expressed as a percentage (see Section 11 for performance criteria for audit accuracy).

6. Interferences and Contamination

6.1 Very volatile compounds, such as chloromethane and vinyl chloride can display peak broadening and co-elution with other species if the compounds are not delivered to the GC column in a small volume of carrier gas. Refocusing of the sample after collection on the primary trap, either on a separate focusing trap or at the head of the gas chromatographic column, mitigates this problem.

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).]

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 (± 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-μm sintered stainless steel in-line filter.

7.1.1.6 **Electronic Timer.** For unattended sample collection.

7.1.1.7 **Solenoid Valve.** Electrically-operated, bi-stable solenoid valve with Viton® seat and O-rings. A Skinner Magnelatch valve is used for purposes of illustration in the text (see Figure 2).

7.1.1.8 **Chromatographic Grade Stainless Steel Tubing and Fittings.** For interconnections. All such materials in contact with sample, analyte, and support gases prior to analysis should be chromatographic grade stainless steel or equivalent.

7.1.1.9 **Thermostatically Controlled Heater.** To maintain above ambient temperature inside insulated sampler enclosure.

7.1.1.10 **Heater Thermostat.** Automatically regulates heater temperature.

7.1.1.11 **Fan.** For cooling sampling system.

7.1.1.12 **Fan Thermostat.** Automatically regulates fan operation.

7.1.1.13 **Maximum-Minimum Thermometer.** Records highest and lowest temperatures during sampling period.

7.1.1.14 **Stainless Steel Shut-off Valve.** Leak free, for vacuum/pressure gauge.

7.1.1.15 **Auxiliary Vacuum Pump.** Continuously draws air through the inlet manifold at 10 L/min. or higher flow rate. Sample is extracted from the manifold at a lower rate, and excess air is exhausted.

[Note: The use of higher inlet flow rates dilutes any contamination present in the inlet and reduces the possibility of sample contamination as a result of contact with active adsorption sites on inlet walls.]

7.1.1.16 **Elapsed Time Meter.** Measures duration of sampling.

7.1.1.17 **Optional Fixed Orifice, Capillary, or Adjustable Micrometering Valve.** May be used in lieu of the electronic flow controller for grab samples or short duration time-integrated samples. Usually appropriate only in situations where screening samples are taken to assess future sampling activity.

7.1.2 Pressurized (see Figure 1 with metal bellows type pump and Figure 3).

7.1.2.1 **Sample Pump.** Stainless steel, metal bellows type, capable of 2 atmospheres output pressure. Pump must be free of leaks, clean, and uncontaminated by oil or organic compounds.

[Note: An alternative sampling system has been developed by Dr. R. Rasmussen, The Oregon Graduate Institute of Science and Technology, 20000 N.W. Walker Rd., Beaverton, Oregon 97006, 503-690-1077, and is illustrated in Figure 3. This flow system uses, in order, a pump, a mechanical flow regulator, and a mechanical compensation flow restrictive device. In this configuration the pump is purged with a large sample flow, thereby eliminating the need for an auxiliary vacuum pump to flush the sample inlet.]

7.1.2.2 **Other Supporting Materials.** All other components of the pressurized sampling system are similar to components discussed in Sections 7.1.1.1 through 7.1.1.17.

7.2 Analytical Apparatus

7.2.1 Sampling/Concentrator System (many commercial alternatives are available).

7.2.1.1 **Electronic Mass Flow Controllers.** Used to maintain constant flow (for purge gas, carrier gas and sample gas) and to provide an analog output to monitor flow anomalies.

7.2.1.2 **Vacuum Pump.** General purpose laboratory pump, capable of reducing the downstream pressure of the flow controller to provide the pressure differential necessary to maintain controlled flow rates of sample air.

7.2.1.3 **Stainless Steel Tubing and Stainless Steel Fittings.** Coated with fused silica to minimize active adsorption sites.

7.2.1.4 **Stainless Steel Cylinder Pressure Regulators.** Standard, two-stage cylinder regulators with pressure gauges.

7.2.1.5 **Gas Purifiers.** Used to remove organic impurities and moisture from gas streams.

7.2.1.6 **Six-port Gas Chromatographic Valve.** For routing sample and carrier gas flows.

7.2.1.7 **Multisorbent Concentrator.** Solid adsorbent packing with various retentive properties for adsorbing trace gases are commercially available from several sources. The packing contains more than one type of adsorbent packed in series.

7.2.1.7.1 A pre-packed adsorbent trap (Supelco 2-0321) containing 200 mg Carbopack B (60/80 mesh) and 50 mg Carbosieve S-III (60/80 mesh) has been found to retain VOCs and allow some water vapor to pass through (6). The addition of a dry purging step allows for further water removal from the adsorbent trap. The steps constituting the dry purge technique that are normally used with multisorbent traps are illustrated in Figure 4. The optimum trapping and dry purging procedure for the Supelco trap consists of a sample volume of 320 mL and a dry nitrogen purge of 1300 mL. Sample trapping and drying is carried out at 25°C. The trap is back-flushed with helium and heated to 220°C to transfer material onto the GC column. A trap bake-out at 260°C for 5 minutes is conducted after each run.

7.2.1.7.2 An example of the effectiveness of dry purging is shown in Figure 5. The multisorbent used in this case is Tenax/Ambersorb 340/Charcoal (7). Approximately 20% of the initial water content in the sample remains after sampling 500 mL of air. The detector response to water vapor (hydrogen atoms detected by atomic emission detection) is plotted versus purge gas volume. Additional water reduction by a factor of 8 is indicated at temperatures of 45°C or higher. Still further water reduction is possible using a two-stage concentration/dryer system.

7.2.1.8 **Cryogenic Concentrator.** Complete units are commercially available from several vendor sources. The characteristics of the latest concentrators include a rapid, "ballistic" heating of the concentrator to release any trapped VOCs into a small carrier gas volume. This facilitates the separation of compounds on the gas chromatographic column.

7.2.2 **Gas Chromatographic/Mass Spectrometric (GC/MS) System.**

7.2.2.1 **Gas Chromatograph.** The gas chromatographic (GC) system must be capable of temperature programming. The column oven can be cooled to subambient temperature (e.g., -50°C) at the start of the gas chromatographic run to effect a resolution of the very volatile organic compounds. In other designs, the rate of release of compounds from the focusing trap in a two stage system obviates the need for retrapping of compounds on the column. The system must include or be interfaced to a concentrator and have all required accessories including analytical columns and gases. All GC carrier gas lines must be constructed from stainless steel or copper tubing. Non-polytetrafluoroethylene (PTFE) thread sealants or flow controllers with Buna-N rubber components must not be used.

7.2.2.2 **Chromatographic Columns.** 100% methyl silicone or 5% phenyl, 95% methyl silicone fused silica capillary columns of 0.25- to 0.53-mm I.D. of varying lengths are recommended for separation of many of the possible subsets of target compounds involving nonpolar compounds. However, considering the diversity of the target list, the choice is left to the operator subject to the performance standards given in Section 11.

7.2.2.3 **Mass Spectrometer.** Either a linear quadrupole or ion trap mass spectrometer can be used as long as it is capable of scanning from 35 to 300 amu every 1 second or less, utilizing 70 volts (nominal) electron energy in the electron impact ionization mode, and producing a mass spectrum which meets all the instrument performance acceptance criteria when 50 ng or less of p-bromofluorobenzene (BFB) is analyzed.

7.2.2.3.1 **Linear Quadrupole Technology.** A simplified diagram of the heart of the quadrupole mass spectrometer is shown in Figure 6. The quadrupole consists of a parallel set of four rod electrodes mounted in a square configuration. The field within the analyzer is created by coupling opposite pairs of rods together and applying radiofrequency (RF) and direct current (DC) potentials between the pairs of rods. Ions created in the ion source from the reaction of column eluates with electrons from the electron source are moved through the parallel array of rods under the influence of the generated field. Ions which are successfully transmitted through the quadrupole are said to possess stable trajectories and are subsequently recorded with the detection system. When the DC potential is zero, a wide band of m/z values is transmitted through the quadrupole. This "RF only" mode is referred to as the "total-ion" mode. In this mode, the quadrupole acts as a strong focusing lens analogous to a high pass filter. The amplitude of the RF determines the low mass cutoff. A mass spectrum is generated by scanning the DC and RF voltages using a fixed DC/RF ratio and a constant drive frequency or by scanning the frequency and holding the DC and RF constant. With the quadrupole system only 0.1 to 0.2 percent of the ions formed in the ion source actually reach the detector.

7.2.2.3.2 **Ion Trap Technology**. An ion-trap mass spectrometer consists of a chamber formed between two metal surfaces in the shape of a hyperboloid of one sheet (ring electrode) and a hyperboloid of two sheets (the two end-cap electrodes). Ions are created within the chamber by electron impact from an electron beam admitted through a small aperture in one of the end caps. Radio frequency (RF) (and sometimes direct current voltage offsets) are applied between the ring electrode and the two end-cap electrodes establishing a quadrupole electric field. This field is uncoupled in three directions so that ion motion can be considered independently in each direction: the force acting upon an ion increases with the displacement of the ion from the center of the field but the direction of the force depends on the instantaneous voltage applied to the ring electrode. A restoring force along one coordinate (such as the distance, r, from the ion-trap's axis of radial symmetry) will exist concurrently with a repelling force along another coordinate (such as the distance, z, along the ion traps axis), and if the field were static the ions would eventually strike an electrode. However, in an RF field the force along each coordinate alternates direction so that a stable trajectory may be possible in which the ions do not strike a surface. In practice, ions of appropriate mass-to-charge ratios may be trapped within the device for periods of milliseconds to hours. A diagram of a typical ion trap is illustrated in Figure 7. Analysis of stored ions is performed by increasing the RF voltage, which makes the ions successively unstable. The effect of the RF voltage on the ring electrode is to "squeeze" the ions in the xy plane so that they move along the z axis. Half the ions are lost to the top cap (held at ground potential); the remaining ions exit the lower end cap to be detected by the electron multiplier. As the energy applied to the ring electrode is increased, the ions are collected in order of increasing mass to produce a conventional mass spectrum. With the ion trap, approximately 50 percent of the generated ions are detected. As a result, a significant increase in sensitivity can be achieved when compared to a full scan linear quadrupole system.

7.2.2.4 **GC/MS Interface**. Any gas chromatograph to mass spectrometer interface that gives acceptable calibration points for each of the analytes of interest and can be used to achieve all acceptable performance criteria may be used. Gas chromatograph to mass spectrometer interfaces constructed of all-glass, glass-lined, or fused silica-lined materials are recommended. Glass and fused silica should be deactivated.

7.2.2.5 **Data System**. The computer system that is interfaced to the mass spectrometer must allow the continuous acquisition and storage, on machine readable media, of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as a Selected Ion Current Profile (SICP). Software must also be available that allows integrating the abundance in any SICP between specified time or scan number limits. Also, software must be available that allows for the comparison of sample spectra with reference library spectra. The National Institute of Standards and Technology (NIST) or Wiley Libraries or equivalent are recommended as reference libraries.

7.2.2.6 **Off-line Data Storage Device**. Device must be capable of rapid recording and retrieval of data and must be suitable for long-term, off-line data storage.

7.3 Calibration System and Manifold Apparatus (see Figure 8)

7.3.1 Calibration Manifold. Stainless steel, glass, or high purity quartz manifold, (e.g., 1.25-cm I.D. x 66-cm) with sampling ports and internal baffles for flow disturbance to ensure proper mixing. The manifold should be heated to ~50°C.

7.3.2 Humidifier. 500-mL impinger flask containing HPLC grade deionized water.

7.3.3 Electronic Mass Flow Controllers. One 0 to 5 L/min unit and one or more 0 to 100 mL/min units for air, depending on number of cylinders in use for calibration.

7.3.4 Teflon Filter(s). 47-mm Teflon® filter for particulate collection.

7.4 Reagents

7.4.1 Neat Materials or Manufacturer-Certified Solutions/Mixtures. Best source (see Section 9).

7.4.2 Helium and Air. Ultra-high purity grade in gas cylinders. He is used as carrier gas in the GC.

7.4.3 Liquid Nitrogen or Liquid Carbon Dioxide. Used to cool secondary trap.

7.4.4 Deionized Water. High performance liquid chromatography (HPLC) grade, ultra-high purity (for humidifier).

8. Collection of Samples in Canisters

8.1 Introduction

8.1.1 Canister samplers, sampling procedures, and canister cleaning procedures have not changed very much from the description given in the original Compendium Method TO-14. Much of the material in this section is therefore simply a restatement of the material given in Compendium Method TO-14, repeated here in order to have all the relevant information in one place.

8.1.2 Recent notable additions to the canister technology has been in the application of canister-based systems for example, to microenvironmental monitoring (8), the capture of breath samples (9), and sector sampling to identify emission sources of VOCs (10).

8.1.3 EPA has also sponsored the development of a mathematical model to predict the storage stability of arbitrary mixtures of trace gases in humidified air (3), and the investigation of the SilcoSteel™ process of coating the canister interior with a film of fused silica to reduce surface activity (11). A recent summary of storage stability data for VOCs in canisters is given in the open literature (5).

8.2 Sampling System Description

8.2.1 Subatmospheric Pressure Sampling [see Figure 1 (without metal bellows type pump)].

8.2.1.1 In preparation for subatmospheric sample collection in a canister, the canister is evacuated to 0.05 mm Hg (see Appendix C for discussion of evacuation pressure). When the canister is opened to the atmosphere containing the VOCs to be sampled, the differential pressure causes the sample to flow into the canister. This technique may be used to collect grab samples (duration of 10 to 30 seconds) or time-weighted-average (TWA) samples (duration of 1-24 hours) taken through a flow-restrictive inlet (e.g., mass flow controller, critical orifice).

8.2.1.2 With a critical orifice flow restrictor, there will be a decrease in the flow rate as the pressure approaches atmospheric. However, with a mass flow controller, the subatmospheric sampling system can maintain a constant flow rate from full vacuum to within about 7 kPa (1.0 psi) or less below ambient pressure.

8.2.2 Pressurized Sampling [see Figure 1 (with metal bellows type pump)].

8.2.2.1 Pressurized sampling is used when longer-term integrated samples or higher volume samples are required. The sample is collected in a canister using a pump and flow control arrangement to achieve a typical 101-202 kPa (15-30 psig) final canister pressure. For example, a 6-liter evacuated canister can be filled at 10 mL/min for 24 hours to achieve a final pressure of 144 kPa (21 psig).

8.2.2.2 In pressurized canister sampling, a metal bellows type pump draws in air from the sampling manifold to fill and pressurize the sample canister.

8.2.3 All Samplers.

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

$$F = \frac{P \times V}{T \times 60}$$

where:

F = flow rate, mL/min.
P = final canister pressure, atmospheres absolute. P is approximately equal to

$$\frac{\text{kPa gauge}}{101.2} - 1$$

V = volume of the canister, mL.
T = sample period, hours.

For example, if a 6-L canister is to be filled to 202 kPa (2 atmospheres) absolute pressure in 24 hours, the flow rate can be calculated by:

$$F = \frac{2 \times 6000}{24 \times 60} = 8.3 \text{ mL/min}$$

8.2.3.2 For automatic operation, the timer is designed to start and stop the pump at appropriate times for the desired sample period. The timer must also control the solenoid valve, to open the valve when starting the pump and to close the valve when stopping the pump.

8.2.3.3 The use of the Skinner Magnelatch valve (see Figure 2) avoids any substantial temperature rise that would occur with a conventional, normally closed solenoid valve that would have to be energized during the entire sample period. The temperature rise in the valve could cause outgassing of organic compounds from the Viton® valve seat material. The Skinner Magnelatch valve requires only a brief electrical pulse to open or close at the appropriate start and stop times and therefore experiences no temperature increase. The pulses may be obtained either with an electronic timer that can be programmed for short (5 to 60 seconds) ON periods, or with a conventional mechanical timer and a special pulse circuit. A simple electrical pulse circuit for operating the Skinner Magnelatch solenoid valve with a conventional mechanical timer is illustrated in Figure 2(a). However, with this simple circuit, the valve may operate unreliably during brief power interruptions or if the timer is manually switched on and off too fast. A better circuit incorporating a time-delay relay to provide more reliable valve operation is shown in Figure 2(b).

8.2.3.4 The connecting lines between the sample inlet and the canister should be as short as possible to minimize their volume. The flow rate into the canister should remain relatively constant over the entire sampling period.

8.2.3.5 As an option, a second electronic timer may be used to start the auxiliary pump several hours prior to the sampling period to flush and condition the inlet line.

8.2.3.6 Prior to field use, each sampling system must pass a humid zero air certification (see Section 8.4.3). All plumbing should be checked carefully for leaks. The canisters must also pass a humid zero air certification before use (see Section 8.4.1).

8.3 Sampling Procedure

8.3.1 The sample canister should be cleaned and tested according to the procedure in Section 8.4.1.

8.3.2 A sample collection system is assembled as shown in Figures 1 and 3 and must be cleaned according to the procedure outlined in Sections 8.4.2 and 8.4.4.

[Note: The sampling system should be contained in an appropriate enclosure.]

8.3.3 Prior to locating the sampling system, the user may want to perform "screening analyses" using a portable GC system, as outlined in Appendix B of Compendium Method TO-14A, to determine potential volatile organics present and potential "hot spots." The information gathered from the portable GC screening analysis would be used in developing a monitoring protocol, which includes the sampling system location, based upon the "screening analysis" results.

8.3.4 After "screening analysis," the sampling system is located. Temperatures of ambient air and sampler box interior are recorded on the canister sampling field test data sheet (FTDS), as documented in Figure 9.

[Note: The following discussion is related to Figure 1]

8.3.5 To verify correct sample flow, a "practice" (evacuated) canister is used in the sampling system.

[Note: For a subatmospheric sampler, a flow meter and practice canister are needed. For the pump-driven system, the practice canister is not needed, as the flow can be measured at the outlet of the system.]

A certified mass flow meter is attached to the inlet line of the manifold, just in front of the filter. The canister is opened. The sampler is turned on and the reading of the certified mass flow meter is compared to the sampler mass flow controller. The values should agree within ±10%. If not, the sampler mass flow meter needs to be recalibrated or there is a leak in the system. This should be investigated and corrected.

[Note: Mass flow meter readings may drift. Check the zero reading carefully and add or subtract the zero reading when reading or adjusting the sampler flow rate to compensate for any zero drift.]

After 2 minutes, the desired canister flow rate is adjusted to the proper value (as indicated by the certified mass flow meter) by the sampler flow control unit controller (e.g., 3.5 mL/min for 24 hr, 7.0 mL/min for 12 hr). Record final flow under "CANISTER FLOW RATE" on the FTDS.

8.3.6 The sampler is turned off and the elapsed time meter is reset to 000.0.

[Note: Whenever the sampler is turned off, wait at least 30 seconds to turn the sampler back on.]

8.3.7 The "practice" canister and certified mass flow meter are disconnected and a clean certified (see Section 8.4.1) canister is attached to the system.

8.3.8 The canister valve and vacuum/pressure gauge valve are opened.

8.3.9 Pressure/vacuum in the canister is recorded on the canister FTDS (see Figure 9) as indicated by the sampler vacuum/pressure gauge.

8.3.10 The vacuum/pressure gauge valve is closed and the maximum-minimum thermometer is reset to current temperature. Time of day and elapsed time meter readings are recorded on the canister FTDS.

8.3.11 The electronic timer is set to start and stop the sampling period at the appropriate times. Sampling starts and stops by the programmed electronic timer.

8.3.12 After the desired sampling period, the maximum, minimum, current interior temperature and current ambient temperature are recorded on the FTDS. The current reading from the flow controller is recorded.

8.3.13 At the end of the sampling period, the vacuum/pressure gauge valve on the sampler is briefly opened and closed and the pressure/vacuum is recorded on the FTDS. Pressure should be close to desired pressure.

[Note: For a subatmospheric sampling system, if the canister is at atmospheric pressure when the field final pressure check is performed, the sampling period may be suspect. This information should be noted on the sampling field data sheet.]

Time of day and elapsed time meter readings are also recorded.

8.3.14 The canister valve is closed. The sampling line is disconnected from the canister and the canister is removed from the system. For a subatmospheric system, a certified mass flow meter is once again connected to the inlet manifold in front of the in-line filter and a "practice" canister is attached to the Magnelatch valve of the sampling system. The final flow rate is recorded on the canister FTDS (see Figure 9).

[Note: For a pressurized system, the final flow may be measured directly.]

The sampler is turned off.

8.3.15 An identification tag is attached to the canister. Canister serial number, sample number, location, and date, as a minimum, are recorded on the tag. The canister is routinely transported back to the analytical laboratory with other canisters in a canister shipping case.

8.4 Cleaning and Certification Program

8.4.1 Canister Cleaning and Certification.

8.4.1.1 All canisters must be clean and free of any contaminants before sample collection.

8.4.1.2 All canisters are leak tested by pressurizing them to approximately 206 kPa (30 psig) with zero air.

[Note: The canister cleaning system in Figure 10 can be used for this task.]

The initial pressure is measured, the canister valve is closed, and the final pressure is checked after 24 hours. If acceptable, the pressure should not vary more than ± 13.8 kPa (± 2 psig) over the 24 hour period.

8.4.1.3 A canister cleaning system may be assembled as illustrated in Figure 10. Cryogen is added to both the vacuum pump and zero air supply traps. The canister(s) are connected to the manifold. The vent shut-off valve and the canister valve(s) are opened to release any remaining pressure in the canister(s). The vacuum pump is started and the vent shut-off valve is then closed and the vacuum shut-off valve is opened. The canister(s) are evacuated to <0.05 mm Hg (see Appendix B) for at least 1 hour.

[Note: On a daily basis or more often if necessary, the cryogenic traps should be purged with zero air to remove any trapped water from previous canister cleaning cycles.]

Air released/evacuated from canisters should be diverted to a fume hood.

8.4.1.4 The vacuum and vacuum/pressure gauge shut-off valves are closed and the zero air shut-off valve is opened to pressurize the canister(s) with humid zero air to approximately 206 kPa (30 psig). If a zero gas generator system is used, the flow rate may need to be limited to maintain the zero air quality.

8.4.1.5 The zero air shut-off valve is closed and the canister(s) is allowed to vent down to atmospheric pressure through the vent shut-off valve. The vent shut-off valve is closed. Repeat Sections 8.4.1.3 through 8.4.1.5 two additional times for a total of three (3) evacuation/pressurization cycles for each set of canisters.

8.4.1.6 At the end of the evacuation/pressurization cycle, the canister is pressurized to 206 kPa (30 psig) with humid zero air. The canister is then analyzed by a GC/MS analytical system. Any canister that has not tested clean (compared to direct analysis of humidified zero air of less than 0.2 ppbv of targeted VOCs) should not be used. As a "blank" check of the canister(s) and cleanup procedure, the final humid zero air fill of 100% of the canisters is analyzed until the cleanup system and canisters are proven reliable (less than 0.2 ppbv of any target VOCs). The check can then be reduced to a lower percentage of canisters.

8.4.1.7 The canister is reattached to the cleaning manifold and is then reevacuated to <0.05 mm Hg (see Appendix B) and remains in this condition until used. The canister valve is closed. The canister is removed from the cleaning system and the canister connection is capped with a stainless steel fitting. The canister is now ready for collection of an air sample. An identification tag is attached to the inlet of each canister for field notes and chain-of-custody purposes. An alternative to evacuating the canister at this point is to store the canisters and reevacuate them just prior to the next use.

8.4.1.8 As an option to the humid zero air cleaning procedures, the canisters are heated in an isothermal oven not to exceed 100°C during evacuation of the canister to ensure that higher molecular weight compounds are not retained on the walls of the canister.

[Note: For sampling more complex VOC mixtures the canisters should be heated to higher temperatures during the cleaning procedure although a special high temperature valve would be needed].

Once heated, the canisters are evacuated to <0.05 mm Hg (see Appendix B) and maintained there for 1 hour. At the end of the heated/evacuated cycle, the canisters are pressurized with humid zero air and analyzed by a GC/MS system after a minimum of 12 hrs of "aging." Any canister that has not tested clean (less than 0.2 ppbv each of targeted compounds) should not be used. Once tested clean, the canisters are reevacuated to <0.05 mm Hg (see Appendix B) and remain in the evacuated state until used. As noted in Section 8.4.1.7, reevacuation can occur just prior to the next use.

8.4.2 Cleaning Sampling System Components.

8.4.2.1 Sample components are disassembled and cleaned before the sampler is assembled. Nonmetallic parts are rinsed with HPLC grade deionized water and dried in a vacuum oven at 50°C. Typically, stainless steel parts and fittings are cleaned by placing them in a beaker of methanol in an ultrasonic bath for 15 minutes. This procedure is repeated with hexane as the solvent.

8.4.2.2 The parts are then rinsed with HPLC grade deionized water and dried in a vacuum oven at 100°C for 12 to 24 hours.

8.4.2.3 Once the sampler is assembled, the entire system is purged with humid zero air for 24 hours.

8.4.3 Zero Air Certification.

[Note: In the following sections, "certification" is defined as evaluating the sampling system with humid zero air and humid calibration gases that pass through all active components of the sampling system. The system is "certified" if no significant additions or deletions (less than 0.2 ppbv each of target compounds) have occurred when challenged with the test gas stream.]

8.4.3.1 The cleanliness of the sampling system is determined by testing the sampler with humid zero air without an evacuated gas sampling canister, as follows.

8.4.3.2 The calibration system and manifold are assembled, as illustrated in Figure 8. The sampler (without an evacuated gas canister) is connected to the manifold and the zero air cylinder is activated to generate a humid gas stream (2 L/min) to the calibration manifold [see Figure 8(b)].

8.4.3.3 The humid zero gas stream passes through the calibration manifold, through the sampling system (without an evacuated canister) to the water management system/VOC preconcentrator of an analytical system.

[Note: The exit of the sampling system (without the canister) replaces the canister in Figure 11.]

After the sample volume (e.g., 500 mL) is preconcentrated on the trap, the trap is heated and the VOCs are thermally desorbed and refocussed on a cold trap. This trap is heated and the VOCs are thermally desorbed onto the head of the capillary column. The VOCs are refocussed prior to gas chromatographic separation. Then, the oven temperature (programmed) increases and the VOCs begin to elute and are detected by a GC/MS (see Section 10) system. The analytical system should not detect greater than 0.2 ppbv of any targeted VOCs in order for the sampling system to pass the humid zero air certification test. Chromatograms (using an FID) of a certified sampler and contaminated sampler are illustrated in Figures 12(a) and 12(b), respectively. If the sampler passes the humid zero air test, it is then tested with humid calibration gas standards containing selected VOCs at concentration levels expected in field sampling (e.g., 0.5 to 2 ppbv) as outlined in Section 8.4.4.

8.4.4 Sampler System Certification with Humid Calibration Gas Standards from a Dynamic Calibration System

8.4.4.1 Assemble the dynamic calibration system and manifold as illustrated in Figure 8.

8.4.4.2 Verify that the calibration system is clean (less than 0.2 ppbv of any target compounds) by sampling a humidified gas stream, *without* gas calibration standards, with a previously certified clean canister (see Section 8.1).

8.4.4.3 The assembled dynamic calibration system is certified clean if less than 0.2 ppbv of any targeted compounds is found.

8.4.4.4 For generating the humidified calibration standards, the calibration gas cylinder(s) containing nominal concentrations of 10 ppmv in nitrogen of selected VOCs is attached to the calibration system as illustrated in Figure 8. The gas cylinders are opened and the gas mixtures are passed through 0 to 10 mL/min certified mass flow controllers to generate ppb levels of calibration standards.

8.4.4.5 After the appropriate equilibrium period, attach the sampling system (containing a certified evacuated canister) to the manifold, as illustrated in Figure 8(b).

8.4.4.6 Sample the dynamic calibration gas stream with the sampling system.

8.4.4.7 Concurrent with the sampling system operation, realtime monitoring of the calibration gas stream is accomplished by the on-line GC/MS analytical system [Figure 8(a)] to provide reference concentrations of generated VOCs.

8.4.4.8 At the end of the sampling period (normally the same time period used for experiments), the sampling system canister is analyzed and compared to the reference GC/MS analytical system to determine if the concentration of the targeted VOCs was increased or decreased by the sampling system.

8.4.4.9 A recovery of between 90% and 110% is expected for all targeted VOCs.

8.4.5 Sampler System Certification without Compressed Gas Cylinder Standards.

8.4.5.1 Not all the gases on the Title III list are available/compatible with compressed gas standards. In these cases sampler certification must be approached by different means.

8.4.5.2 Definitive guidance is not currently available in these cases; however, Section 9.2 lists several ways to generate gas standards. In general, Compendium Method TO-14A compounds (see Table 1) are available commercially as compressed gas standards.

9. GC/MS Analysis of Volatiles from Canisters

9.1 Introduction

9.1.1 The analysis of canister samples is accomplished with a GC/MS system. Fused silica capillary columns are used to achieve high temporal resolution of target compounds. Linear quadrupole or ion trap mass spectrometers are employed for compound detection. The heart of the system is composed of the sample inlet concentrating device that is needed to increase sample loading into a detectable range. Two examples of concentrating systems are discussed. Other approaches are acceptable as long as they are compatible with achieving the system performance criteria given in Section 11.

9.1.2 With the first technique, a whole air sample from the canister is passed through a multisorbent packing (including single adsorbent packings) contained within a metal or glass tube maintained at or above the surrounding air temperature. Depending on the water retention properties of the packing, some or most of the water vapor passes completely through the trap during sampling. Additional drying of the sample is accomplished after the sample concentration is completed by forward purging the trap with clean, dry helium or another inert gas (air is not used). The sample is then thermally desorbed from the packing and backflushed from the trap onto a gas chromatographic column. In some systems a "refocusing" trap is placed between the primary trap and the gas chromatographic column. The specific system design downstream of the primary trap depends on technical factors such as the rate of thermal desorption and sampled volume, but the objective in most cases is to enhance chromatographic resolution of the individual sample components before detection on a mass spectrometer.

9.1.3 Sample drying strategies depend on the target list of compounds. For some target compound lists, the multisorbent packing of the concentrator can be selected from hydrophobic adsorbents which allow a high percentage of water vapor in the sample to pass through the concentrator during sampling and without significant loss of the target compounds. However, if very volatile organic compounds are on the target list, the adsorbents required for their retention may also strongly retain water vapor and a more lengthy dry purge is necessary prior to analysis.

9.1.4 With the second technique, a whole air sample is passed through a concentrator where the VOCs are condensed on a reduced temperature surface (cold trap). Subsequently, the condensed gases are thermally desorbed and backflushed from the trap with an inert gas onto a gas chromatographic column. This concentration technique is similar to that discussed in Compendium Method TO-14, although a membrane dryer is not used. The sample size is reduced in volume to limit the amount of water vapor that is also collected (100 mL or less may be necessary). The attendant reduction in sensitivity is offset by enhancing the sensitivity of detection, for example by using an ion trap detector.

9.2 Preparation of Standards

9.2.1 Introduction.

9.2.1.1 When available, standard mixtures of target gases in high pressure cylinders must be certified traceable to a NIST Standard Reference Material (SRM) or to a NIST/EPA approved Certified Reference Material (CRM). Manufacturer's certificates of analysis must be retained to track the expiration date.

9.2.1.2 The neat standards that are used for making trace gas standards must be of high purity; generally a purity of 98 percent or better is commercially available.

9.2.1.3 Cylinder(s) containing approximately 10 ppmv of each of the target compounds are typically used as primary stock standards. The components may be purchased in one cylinder or in separate cylinders depending on compatibility of the compounds and the pressure of the mixture in the cylinder. Refer to manufacturer's specifications for guidance on purchasing and mixing VOCs in gas cylinders.

9.2.2 Preparing Working Standards.

9.2.2.1 **Instrument Performance Check Standard.** Prepare a standard solution of BFB in humidified zero air at a concentration which will allow collection of 50 ng of BFB or less under the optimized concentration parameters.

9.2.2.2 **Calibration Standards.** Prepare five working calibration standards in humidified zero air at a concentration which will allow collection at the 2, 5, 10, 20, and 50 ppbv level for each component under the optimized concentration parameters.

9.2.2.3 **Internal Standard Spiking Mixture.** Prepare an internal spiking mixture containing bromochloromethane, chlorobenzene-d_5, and 1,4-difluorobenzene at 10 ppmv each in humidified zero air to be added to the sample or calibration standard. 500 µL of this mixture spiked into 500 mL of sample will result in a concentration of 10 ppbv. The internal standard is introduced into the trap during the collection time for all calibration, blank, and sample analyses using the apparatus shown in Figure 13 or by equivalent means. The volume of internal standard spiking mixture added for each analysis must be the same from run to run.

9.2.3 Standard Preparation by Dynamic Dilution Technique.

9.2.3.1 Standards may be prepared by dynamic dilution of the gaseous contents of a cylinder(s) containing the gas calibration stock standards with humidified zero air using mass flow controllers and a calibration manifold. The working standard may be delivered from the manifold to a clean, evacuated canister using a pump and mass flow controller.

9.2.3.2 Alternatively, the analytical system may be calibrated by sampling directly from the manifold if the flow rates are optimized to provide the desired amount of calibration standards. However, the use of the canister as a reservoir prior to introduction into the concentration system resembles the procedure normally used to collect samples and is preferred. Flow rates of the dilution air and cylinder standards (all expressed in the same units) are measured using a bubble meter or calibrated electronic flow measuring device, and the concentrations of target compounds in the manifold are then calculated using the dilution ratio and the original concentration of each compound.

$$\text{Manifold Conc.} = \frac{(\text{Original Conc.})\ (\text{Std. Gas Flowrate})}{(\text{Air Flowrate}) + (\text{Std. Gas Flowrate})}$$

9.2.3.3 Consider the example of 1 mL/min flow of 10 ppmv standard diluted with 1,000 mL/min of humid air provides a nominal 10 ppbv mixture, as calculated below:

$$\text{Manifold Conc.} = \frac{(10\ \text{ppm})(1\ \text{mL/min})(1000\ \text{ppb/1 ppm})}{(1000\ \text{mL/min}) + (1\ \text{mL/min})} = 10\ \text{ppb}$$

9.2.4 Standard Preparation by Static Dilution Bottle Technique

[Note: Standards may be prepared in canisters by spiking the canister with a mixture of components prepared in a static dilution bottle (12). This technique is used specifically for liquid standards.]

9.2.4.1 The volume of a clean 2-liter round-bottom flask, modified with a threaded glass neck to accept a Mininert septum cap, is determined by weighing the amount of water required to completely fill up the flask. Assuming a density for the water of 1 g/mL, the weight of the water in grams is taken as the volume of the flask in milliliters.

9.2.4.2 The flask is flushed with helium by attaching a tubing into the glass neck to deliver the helium. After a few minutes, the tubing is removed and the glass neck is immediately closed with a Mininert septum cap.

9.2.4.3 The flask is placed in a 60°C oven and allowed to equilibrate at that temperature for about 15 minutes. Predetermined aliquots of liquid standards are injected into the flask making sure to keep the flask temperature constant at 60°C.

9.2.4.4 The contents are allowed to equilibrate in the oven for at least 30 minutes. To avoid condensation, syringes must be preheated in the oven at the same temperature prior to withdrawal of aliquots to avoid condensation.

9.2.4.5 Sample aliquots may then be taken for introduction into the analytical system or for further dilution. An aliquot or aliquots totaling greater than 1 percent of the flask volume should be avoided.

9.2.4.6 Standards prepared by this method are stable for one week. The septum must be replaced with each freshly prepared standard.

9.2.4.7 The concentration of each component in the flask is calculated using the following equation:

$$\text{Concentration, mg/L} = \frac{(V_a)(d)}{V_f}$$

where:

V_a = Volume of liquid neat standard injected into the flask, µL.

d = Density of the liquid neat standard, mg/µL.

V_f = Volume of the flask, L.

9.2.4.8 To obtain concentrations in ppbv, the equation given in Section 9.2.5.7 can be used.

[Note: In the preparation of standards by this technique, the analyst should make sure that the volume of neat standard injected into the flask does not result in an overpressure due to the higher partial pressure produced by the standard compared to the vapor pressure in the flask. Precautions should also be taken to avoid a significant decrease in pressure inside the flask after withdrawal of aliquot(s).]

9.2.5 Standard Preparation Procedure in High Pressure Cylinders

[Note: Standards may be prepared in high pressure cylinders (13). A modified summary of the procedure is provided below.]

9.2.5.1 The standard compounds are obtained as gases or neat liquids (greater than 98 percent purity).

9.2.5.2 An aluminum cylinder is flushed with high-purity nitrogen gas and then evacuated to better than 25 in. Hg.

9.2.5.3 Predetermined amounts of each neat standard compound are measured using a microliter or gastight syringe and injected into the cylinder. The cylinder is equipped with a heated injection port and nitrogen flow to facilitate sample transfer.

9.2.5.4 The cylinder is pressurized to 1000 psig with zero nitrogen.

[Note: User should read all SOPs associated with generating standards in high pressure cylinders. Follow all safety requirements to minimize danger from high pressure cylinders.]

9.2.5.5 The contents of the cylinder are allowed to equilibrate (~24 hrs) prior to withdrawal of aliquots into the GC system.

9.2.5.6 If the neat standard is a gas, the cylinder concentration is determined using the following equation:

$$\text{Concentration, ppbv} = \frac{\text{Volume}_{\text{standard}}}{\text{Volume}_{\text{dilution gas}}} \times 10^9$$

[Note: Both values must be expressed in the same units.]

9.2.5.7 If the neat standard is a liquid, the gaseous concentration can be determined using the following equations:

$$V = \frac{nRT}{P}$$

and:

$$n = \frac{(mL)(d)}{MW}$$

where:

V = Gaseous volume of injected compound at EPA standard temperature (25°C) and pressure (760 mm Hg), L.
n = Moles.
R = Gas constant, 0.08206 L-atm/mole °K.
T = 298°K (standard temperature).
P = 1 standard pressure, 760 mm Hg (1 atm).
mL = Volume of liquid injected, mL.
d = Density of the neat standard, g/mL.
MW = Molecular weight of the neat standard expressed, g/g-mole.

The gaseous volume of the injected compound is divided by the cylinder volume at STP and then multiplied by 10^9 to obtain the component concentration in ppb units.

9.2.6 Standard Preparation by Water Methods.

[Note: Standards may be prepared by a water purge and trap method (14) and summarized as follows].

9.2.6.1 A previously cleaned and evacuated canister is pressurized to 760 mm Hg absolute (1 atm) with zero grade air.

9.2.6.2 The air gauge is removed from the canister and the sparging vessel is connected to the canister with the short length of 1/16 in. stainless steel tubing.

[Note: Extra effort should be made to minimize possible areas of dead volume to maximize transfer of analytes from the water to the canister.]

9.2.6.3 A measured amount of the stock standard solution and the internal standard solution is spiked into 5 mL of water.

9.2.6.4 This water is transferred into the sparge vessel and purged with nitrogen for 10 mins at 100 mL/min. The sparging vessel is maintained at 40°C.

9.2.6.5 At the end of 10 mins. the sparge vessel is removed and the air gauge is re-installed, to further pressurize the canister with pure nitrogen to 1500 mm Hg absolute pressure (approximately 29 psia).

9.2.6.6 The canister is allowed to equilibrate overnight before use.

9.2.6.7 A schematic of this approach is shown in Figure 14.

9.2.7 Preparation of Standards by Permeation Tubes.

9.2.7.1 Permeation tubes can be used to provide standard concentration of a trace gas or gases. The permeation of the gas can occur from inside a permeation tube containing the trace species of interest to an air stream outside. Permeation can also occur from outside a permeable membrane tube to an air stream passing through the tube (e.g., a tube of permeable material immersed in a liquid).

9.2.7.2 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 over 250 compounds. Some commercial suppliers of permeation tube equipment are listed in Appendix D.

9.2.8 Storage of Standards.

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

9.2.8.2 It is imperative that a storage logbook be kept to document storage time.

10. GC/MS Operating Conditions

10.1 Preconcentrator

The following are typical cryogenic and adsorbent preconcentrator analytical conditions which, however, depend on the specific combination of solid sorbent and must be selected carefully by the operator. The reader is referred to Tables 1 and 2 of Compendium Method TO-17 for guidance on selection of sorbents. An example of a system using a solid adsorbent preconcentrator with a cryofocusing trap is discussed in the literature (15). Oven temperature programming starts above ambient.

10.1.1 Sample Collection Conditions

Cryogenic Trap Adsorbent Trap

Set point	-150°C	Set point	27°C
Sample volume	- up to 100 mL	Sample volume	- up to 1,000 mL
Carrier gas purge flow	- none	Carrier gas purge flow	- selectable

[Note: The analyst should optimize the flow rate, duration of sampling, and absolute sample volume to be used. Other preconcentration systems may be used provided performance standards (see Section 11) are realized.]

10.1.2 Desorption Conditions

Cryogenic Trap		Adsorbent Trap	
Desorb Temperature	120°C	Desorb Temperature	Variable
Desorb Flow Rate	~ 3 mL/min He	Desorb Flow Rate	~3 mL/min He
Desorb Time	<60 sec	Desorb Time	<60 sec

The adsorbent trap conditions depend on the specific solid adsorbents chosen (see manufacturers' specifications).

10.1.3 Trap Reconditioning Conditions.

Cryogenic Trap		Adsorbent Trap	
Initial bakeout	120°C (24 hrs)	Initial bakeout	
Variable (24 hrs)			
After each run	120°C (5 min)	After each run	Variable (5 min)

10.2 GC/MS System

10.2.1 Optimize GC conditions for compound separation and sensitivity. Baseline separation of benzene and carbon tetrachloride on a 100% methyl polysiloxane stationary phase is an indication of acceptable chromatographic performance.

10.2.2 The following are the recommended gas chromatographic analytical conditions when using a 50-meter by 0.3-mm I.D., 1 µm film thickness fused silica column with refocusing on the column.

Item	Condition	
Carrier Gas:	Helium	
Flow Rate:	Generally 1-3 mL/min as recommended by manufacturer	
Temperature Program:	Initial Temperature:	-50°C
	Initial Hold Time:	2 min
	Ramp Rate:	8° C/min
	Final Temperature:	200°C
	Final Hold Time:	Until all target compounds elute.

10.2.3 The following are the recommended mass spectrometer conditions:

Item	Condition

Electron Energy: 70 Volts (nominal)
Mass Range: 35-300 amu [the choice of 35 amu excludes the detection of some target compounds such as methanol and formaldehyde, and the quantitation of others such as ethylene oxide, ethyl carbamate, etc. (see Table 2). Lowering the mass range and using special programming features available on modern gas chromatographs will be necessary in these cases, but are not considered here.
Scan Time: To give at least 10 scans per peak, not to exceed 1 second per scan].

A schematic for a typical GC/MS analytical system is illustrated in Figure 15.

10.3 Analytical Sequence

10.3.1 **Introduction.** The recommended GC/MS analytical sequence for samples during each 24-hour time period is as follows:

- Perform instrument performance check using bromofluorobenzene (BFB).
- Initiate multi-point calibration or daily calibration checks.
- Perform a laboratory method blank.
- Complete this sequence for analysis of ≤20 field samples.

10.4 Instrument Performance Check

10.4.1 **Summary.** It is necessary to establish that a given GC/MS meets tuning and standard mass spectral abundance criteria prior to initiating any data collection. The GC/MS system is set up according to the manufacturer's specifications, and the mass calibration and resolution of the GC/MS system are then verified by the analysis of the instrument performance check standard, bromofluorobenzene (BFB).

10.4.2 **Frequency.** Prior to the analyses of any samples, blanks, or calibration standards, the Laboratory must establish that the GC/MS system meets the mass spectral ion abundance criteria for the instrument performance check standard containing BFB. The instrument performance check solution must be analyzed initially and once per 24-hour time period of operation.

The 24-hour time period for GC/MS instrument performance check and standards calibration (initial calibration or daily calibration check criteria) begins at the injection of the BFB which the laboratory records as documentation of a compliance tune.

10.4.3 **Procedure.** The analysis of the instrument performance check standard is performed by trapping 50 ng of BFB under the optimized preconcentration parameters. The BFB is introduced from a cylinder into the GC/MS via a sample loop valve injection system similar to that shown in Figure 13.

The mass spectrum of BFB must be acquired in the following manner. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is conducted using a single scan prior to the elution of BFB.

10.4.4 **Technical Acceptance Criteria.** Prior to the analysis of any samples, blanks, or calibration standards, the analyst must establish that the GC/MS system meets the mass spectral ion abundance criteria for the instrument performance check standard as specified in Table 3.

10.4.5 **Corrective Action.** If the BFB acceptance criteria are not met, the MS must be retuned. It may be necessary to clean the ion source, or quadrupoles, or take other necessary actions to achieve the acceptance criteria.

10.4.6 Documentation. Results of the BFB tuning are to be recorded and maintained as part of the instrumentation log.

10.5 Initial Calibration

10.5.1 Summary. Prior to the analysis of samples and blanks but after the instrument performance check standard criteria have been met, each GC/MS system must be calibrated at five concentrations that span the monitoring range of interest in an initial calibration sequence to determine instrument sensitivity and the linearity of GC/MS response for the target compounds. For example, the range of interest may be 2 to 20 ppbv, in which case the five concentrations would be 1, 2, 5, 10 and 25 ppbv.

One of the calibration points from the initial calibration curve must be at the same concentration as the daily calibration standard (e.g., 10 ppbv).

10.5.2 Frequency. Each GC/MS system must be recalibrated following corrective action (e.g., ion source cleaning or repair, column replacement, etc.) which may change or affect the initial calibration criteria or if the daily calibration acceptance criteria have not been met.

If time remains in the 24-hour time period after meeting the acceptance criteria for the initial calibration, samples may be analyzed.

If time does not remain in the 24-hour period after meeting the acceptance criteria for the initial calibration, a new analytical sequence shall commence with the analysis of the instrument performance check standard followed by analysis of a daily calibration standard.

10.5.3 Procedure. Verify that the GC/MS system meets the instrument performance criteria in Section 10.4.

The GC must be operated using temperature and flow rate parameters equivalent to those in Section 10.2.2. Calibrate the preconcentration-GC/MS system by drawing the standard into the system. Use one of the standards preparation techniques described under Section 9.2 or equivalent.

A minimum of five concentration levels are needed to determine the instrument sensitivity and linearity. One of the calibration levels should be near the detection level for the compounds of interest. The calibration range should be chosen so that linear results are obtained as defined in Sections 10.5.1 and 10.5.5.

Quantitation ions for the target compounds are shown in Table 2. The primary ion should be used unless interferences are present, in which case a secondary ion is used.

10.5.4 Calculations.

[Note: In the following calculations, an internal standard approach is used to calculate response factors. The area response used is that of the primary quantitation ion unless otherwise stated.]

10.5.4.1 Relative Response Factor (RRF). Calculate the relative response factors for each target compound relative to the appropriate internal standard (i.e., standard with the nearest retention time) using the following equation:

$$RRF = \frac{A_x C_{is}}{A_{is} C_x}$$

where: RRF = Relative response factor.

A_x = Area of the primary ion for the compound to be measured, counts.

A_{is} = Area of the primary ion for the internal standard, counts.

C_{is} = Concentration of internal standard spiking mixture, ppbv.

C_x = Concentration of the compound in the calibration standard, ppbv.

[Note: The equation above is valid under the condition that the volume of internal standard spiking mixture added in all field and QC analyses is the same from run to run, and that the volume of field and QC sample introduced into the trap is the same for each analysis. C_{is} and C_x must be in the same units.]

10.5.4.2 **Mean Relative Response Factor.** Calculate the mean RRF for each compound by averaging the values obtained at the five concentrations using the following equation:

$$\overline{RRF} = \sum_{i=1}^{n} \frac{x_i}{n}$$

where: $\overline{RRF}$ = Mean relative response factor.

x_i = RRF of the compound at concentration i.

n = Number of concentration values, in this case 5.

10.5.4.3 **Percent Relative Standard Deviation (%RSD).** Using the RRFs from the initial calibration, calculate the %RSD for all target compounds using the following equations:

$$\%RSD = \frac{SD_{RRF}}{\overline{RRF}} \times 100$$

and

$$SD_{RRF} = \sqrt{\sum_{i=1}^{N} \frac{(RRF_i - \overline{RRF})^2}{N - 1}}$$

where: SD_{RRF} = Standard deviation of initial response factors (per compound).

RRF_i = Relative response factor at a concentration level i.

$\overline{RRF}$ = Mean of initial relative response factors (per compound).

10.5.4.4 **Relative Retention Times (RRT).** Calculate the RRTs for each target compound over the initial calibration range using the following equation:

$$RRT = \frac{RT_c}{RT_{is}}$$

where: RT_c = Retention time of the target compound, seconds

RT_{is} = Retention time of the internal standard, seconds.

10.5.4.5 **Mean of the Relative Retention Times ($\overline{RRT}$).** Calculate the mean of the relative retention times ($\overline{RRT}$) for each analyte target compound over the initial calibration range using the following equation:

$$\overline{RRT} = \sum_{i=1}^{n} \frac{RRT}{n}$$

where: $\overline{RRT}$ = Mean relative retention time for the target compound for each initial calibration standard.

RRT = Relative retention time for the target compound at each calibration level.

10.5.4.6 **Tabulate Primary Ion Area Response (Y) for Internal Standard.** Tabulate the area response (Y) of the primary ions (see Table 2) and the corresponding concentration for each compound and internal standard.

10.5.4.7 **Mean Area Response ($\overline{Y}$) for Internal Standard.** Calculate the mean area response ($\overline{Y}$) for each internal standard compound over the initial calibration range using the following equation:

$$\overline{Y} = \sum_{i=1}^{n} \frac{Y_i}{n}$$

where: $\overline{Y}$ = Mean area response.

Y = Area response for the primary quantitation ion for the internal standard for each initial calibration standard.

10.5.4.8 **Mean Retention Times ($\overline{RT}$).** Calculate the mean of the retention times ($\overline{RT}$) for each internal standard over the initial calibration range using the following equation:

$$\overline{RT} = \sum_{i=1}^{n} \frac{RT_i}{n}$$

where: $\overline{RT}$ = Mean retention time, seconds

RT = Retention time for the internal standard for each initial calibration standard, seconds.

10.5.5 **Technical Acceptance Criteria for the Initial Calibration.**

10.5.5.1 The calculated %RSD for the RRF for each compound in the calibration table must be less than 30% with at most two exceptions up to a limit of 40%.

[Note: This exception may not be acceptable for all projects. Many projects may have a specific target list of compounds which would require the lower limit for all compounds.]

10.5.5.2 The RRT for each target compound at each calibration level must be withiin 0.06 RRT units of the mean RRT for the compound.

10.5.5.3 The area response Y of at each calibration level must be within 40% of the mean area response $\overline{Y}$ over the initial calibration range for each internal standard.

10.5.5.4 The retention time shift for each of the internal standards at each calibration level must be within 20 s of the mean retention time over the initial calibration range for each internal standard.

10.5.6 **Corrective Action.**

10.5.6.1 **Criteria.** If the initial calibration technical acceptance criteria are not met, inspect the system for problems. It may be necessary to clean the ion source, change the column, or take other corrective actions to meet the initial calibration technical acceptance criteria.

10.5.6.2 **Schedule.** Initial calibration acceptance criteria <u>must</u> be met before any field samples, performance evaluation (PE) samples, or blanks are analyzed.

10.6 Daily Calibration

10.6.1 Summary. Prior to the analysis of samples and blanks but after tuning criteria have been met, the initial calibration of each GC/MS system must be routinely checked by analyzing a daily calibration standard to ensure that the instrument continues to remain under control. The daily calibration standard, which is the nominal 10 ppbv level calibration standard, should contain all the target compounds.

10.6.2 Frequency. A check of the calibration curve must be performed once every 24 hours on a GC/MS system that has met the tuning criteria. The daily calibration sequence starts with the injection of the BFB. If the BFB analysis meets the ion abundance criteria for BFB, then a daily calibration standard may be analyzed.

10.6.3 Procedure. The mid-level calibration standard (10 ppbv) is analyzed in a GC/MS system that has met the tuning and mass calibration criteria following the same procedure in Section 10.5.

10.6.4 Calculations. Perform the following calculations.

[Note: As indicated earlier, the area response of the primary quantitation ion is used unless otherwise stated.]

10.6.4.1 Relative Response Factor (RRF). Calculate a relative response factor (RRF) for each target compound using the equation in Section 10.5.4.1.

10.6.4.2 Percent Difference (%D). Calculate the percent difference in the RRF of the daily RRF (24-hour) compared to the mean RRF in the most recent initial calibration. Calculate the %D for each target compound using the following equation:

$$\%D = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} \times 100$$

where:

RRF_c = RRF of the compound in the continuing calibration standard.

$\overline{RRF_i}$ = Mean RRF of the compound in the most recent initial calibration.

10.6.5 Technical Acceptance Criteria. The daily calibration standard must be analyzed at the concentration level and frequency described in this Section 10.6 and on a GC/MS system meeting the BFB instrument performance check criteria (see Section 10.4).

The %D for each target compound in a daily calibration sequence must be within ±30 percent in order to proceed with the analysis of samples and blanks. A control chart showing %D values should be maintained.

10.6.6 Corrective Action. If the daily calibration technical acceptance criteria are not met, inspect the system for problems. It may be necessary to clean the ion source, change the column, or take other corrective actions to meet the daily calibration technical acceptance criteria.

Daily calibration acceptance criteria must be met before any field samples, performance evaluation (PE) samples, or blanks are analyzed. If the % D criteria are not met, it will be necessary to rerun the daily calibration sample.

10.7 Blank Analyses

10.7.1 Summary. To monitor for possible laboratory contamination, laboratory method blanks are analyzed at least once in a 24-hour analytical sequence. All steps in the analytical procedure are performed on the blank 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 quantitation 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

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

in humidity, being lower at the higher canister pressures and increasing in humidity as the canister pressures decreases. Storage integrity of water soluble compounds may also be affected.]

10.8.2 **Frequency**. If time remains in the 24-hour period in which an initial calibration is performed, samples may be analyzed without analysis of a daily calibration standard.

If time does not remain in the 24-hour period since the injection of the instrument performance check standard in which an initial calibration is performed, both the instrument performance check standard and the daily calibration standard should be analyzed before sample analysis may begin.

10.8.3 **Procedure for Instrumental Analysis**. Perform the following procedure for analysis.

10.8.3.1 All canister samples should be at temperature equilibrium with the laboratory.

10.8.3.2 Check and adjust the mass flow controllers to provide correct flow rates for the system.

10.8.3.3 Connect the sample canister to the inlet of the GC/MS analytical system, as shown in Figure 15 [Figure 16 shows an alternate two stage concentrator using multisorbent traps followed by a trap cooled by a closed cycle cooler (15)]. The desired sample flow is established through the six-port chromatographic valve and the preconcentrator to the downstream flow controller. The absolute volume of sample being pulled through the trap must be consistent from run to run.

10.8.3.4 Heat/cool the GC oven and cryogenic or adsorbent trap to their set points. Assuming a six-port value is being used, as soon as the trap reaches its lower set point, the six-port chromatographic valve is cycled to the trap position to begin sample collection. Utilize the sample collection time which has been optimized by the analyst.

10.8.3.5 Use the arrangement shown in Figure 13, (i.e., a gastight syringe or some alternate method) introduce an internal standard during the sample collection period. Add sufficient internal standard equivalent to 10 ppbv in the sample. For example, a 0.5 mL volume of a mixture of internal standard compounds, each at 10 ppmv concentration, added to a sample volume of 500 mL, will result in 10 ppbv of each internal standard in the sample.

10.8.3.6 After the sample and internal standards are preconcentrated on the trap, the GC sampling valve is cycled to the inject position and the trap is swept with helium and heated. Assuming a focusing trap is being used, the trapped analytes are thermally desorbed onto a focusing trap and then onto the head of the capillary column and are separated on the column using the GC oven temperature program. The canister valve is closed and the canister is disconnected from the mass flow controller and capped. The trap is maintained at elevated temperature until the beginning of the next analysis.

10.8.3.7 Upon sample injection onto the column, the GC/MS system is operated so that the MS scans the atomic mass range from 35 to 300 amu. At least ten scans per eluting chromatographic peak should be acquired. Scanning also allows identification of unknown compounds in the sample through searching of library spectra.

10.8.3.8 Each analytical run must be checked for saturation. The level at which an individual compound will saturate the detection system is a function of the overall system sensitivity and the mass spectral characteristics of that compound.

10.8.3.9 Secondary ion quantitation is allowed only when there are sample matrix interferences with the primary ion. If secondary ion quantitation is performed, document the reasons in the laboratory record book.

10.8.4 **Calculations**. The equation below is used for calculating concentrations.

$$C_x = \frac{A_x C_{is} DF}{A_{is} \overline{RRF}}$$

where: C_x = Compound concentration, ppbv.

A_x = Area of the characteristic ion for the compound to be measured, counts.

A_{is} = Area of the characteristic ion for the specific internal standard, counts.

C_{is} = Concentration of the internal standard spiking mixture, ppbv

$\overline{RRF}$ = Mean relative response factor from the initial calibration.

DF = Dilution factor calculated as described in section 2. If no dilution is performed, DF = 1.

[Note: The equation above is valid under the condition that the volume (~500 µL) of internal standard spiking mixture added in all field and QC analyses is the same from run to run, and that the volume (~500 mL) of field and QC sample introduced into the trap is the same for each analysis.]

10.8.5 Technical Acceptance Criteria.

[Note: If the most recent valid calibration is an initial calibration, internal standard area responses and RTs in the sample are evaluated against the corresponding internal standard area responses and RTs in the mid level standard (10 ppbv) of the initial calibration.]

10.8.5.1 The field sample must be analyzed on a GC/MS system meeting the BFB tuning, initial calibration, and continuing calibration technical acceptance criteria at the frequency described in Sections 10.4, 10.5 and 10.6.

10.8.5.2 The field samples must be analyzed along with a laboratory method blank that met the blank technical acceptance criteria.

10.8.5.3 All of the target analyte peaks should be within the initial calibration range.

10.8.5.4 The retention time for each internal standard must be within ±0.33 minutes of the retention time of the internal standard in the most recent valid calibration.

10.8.6 Corrective Action. If the on-column concentration of any compound in any sample exceeds the initial calibration range, an aliquot of the original sample must be diluted and reanalyzed. Guidance in performing dilutions and exceptions to this requirement are given below.

- Use the results of the original analysis to determine the approximate dilution factor required to get the largest analyte peak within the initial calibration range.
- The dilution factor chosen should keep the response of the largest analyte peak for a target compound in the upper half of the initial calibration range of the instrument.

[Note: Analysis involving dilution should be reported with a dilution factor and nature of the dilution gas.]

10.8.6.1 Internal standard responses and retention times must be evaluated during or immediately after data acquisition. If the retention time for any internal standard changes by more than 20 sec from the latest daily (24-hour) calibration standard (or mean retention time over the initial calibration range), the GC/MS system must be inspected for malfunctions, and corrections made as required.

10.8.6.2 If the area response for any internal standard changes by more than ±40 percent between the sample and the most recent valid calibration, the GC/MS system must be inspected for malfunction and corrections made as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is necessary.

10.8.6.3 If, after reanalysis, the area responses or the RTs for all internal standards are inside the control limits, then the problem with the first analysis is considered to have been within the control of the Laboratory. Therefore, submit only data from the analysis with SICPs within the limits. This is considered the initial analysis and should be reported as such on all data deliverables.

11. Requirements for Demonstrating Method Acceptability for VOC Analysis from Canisters

11.1 Introduction

11.1.1 There are three performance criteria which must be met for a system to qualify under Compendium Method TO-15. These criteria are: the method detection limit of ≤0.5 ppbv, replicate precision within 25 percent, and audit accuracy within 30 percent for concentrations normally expected in contaminated ambient air (0.5 to 25 ppbv).

11.1.2 Either SIM or SCAN modes of operation can be used to achieve these criteria, and the choice of mode will depend on the number of target compounds, the decision of whether or not to determine tentatively identified compounds along with other VOCs on the target list, as well as on the analytical system characteristics.

11.1.3 Specific criteria for each Title III compound on the target compound list must be met by the analytical system. These criteria were established by examining summary data from EPA's Toxics Air Monitoring System Network and the Urban Air Toxics Monitoring Program network. Details for the determination of each of the criteria follow.

11.2 Method Detection Limit

11.2.1 The procedure chosen to define the method detection limit is that given in the *Code of Federal Regulations* (40 CFR 136 Appendix B).

11.2.2 The method detection limit is defined for each system by making seven replicate measurements of the compound of interest at a concentration near (within a factor of five) the expected detection limit, computing the standard deviation for the seven replicate concentrations, and multiplying this value by 3.14 (i.e., the Student's t value for 99 percent confidence for seven values). Employing this approach, the detection limits given in Table 4 were obtained for some of the VOCs of interest.

11.3 Replicate Precision

11.3.1 The measure of replicate precision used for this program is the absolute value of the difference between replicate measurements of the sample divided by the average value and expressed as a percentage as follows:

$$\text{percent difference} = \frac{|x_1 - x_2|}{\bar{x}} \times 100$$

where:

x_1 = First measurement value.

x_2 = Second measurement value.

$\bar{x}$ = Average of the two values.

11.3.2 There are several factors which may affect the precision of the measurement. The nature of the compound of interest itself such as molecular weight, water solubility, polarizability, etc., each have some effect on the precision, for a given sampling and analytical system. For example, styrene, which is classified as a polar VOC, generally shows slightly poorer precision than the bulk of nonpolar VOCs. A primary influence on precision is the concentration level of the compound of interest in the sample, i.e., the precision degrades as the concentration approaches the detection limit. A conservative measure was obtained from replicate analysis of "real world" canister samples from the TAMS and UATMP networks. These data are summarized in Table 5 and suggest that a replicate precision value of 25 percent can be achieved for each of the target compounds.

11.4 Audit Accuracy

11.4.1 A measure of analytical accuracy is the degree of agreement with audit standards. Audit accuracy is defined as the difference between the nominal concentration of the audit compound and the measured value divided by the audit value and expressed as a percentage, as illustrated in the following equation:

$$\text{Audit Accuracy, \%} = \frac{\text{Spiked Value} - \text{Observed Value}}{\text{Spiked Value}} \times 100$$

11.4.2 Audit accuracy results for TAMS and UATMP analyses are summarized in Table 6 and were used to form the basis for a selection of 30 percent as the performance criterion for audit accuracy.

12. References

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APPENDIX A.

LISTING OF SOME COMMERCIAL WATER MANAGEMENT SYSTEMS USED WITH AUTOGC SYSTEMS

Tekmar Dohrman Company
7143 East Kemper Road
Post Office Box 429576
Cincinnati, Ohio 45242-9576
(513) 247-7000
(513) 247-7050 (Fax)
(800) 543-4461
[Moisture control module]

Entech Laboratory Automation
950 Enchanted Way No. 101
Simi Valley, California 93065
(805) 527-5939
(805) 527-5687 (Fax)
[Microscale Purge and Trap]

Dynatherm Analytical Instruments
Post Office Box 159
Kelton, Pennsylvania 19346
(215) 869-8702
(215) 869-3885 (Fax)
[Thermal Desorption System]

XonTech Inc.
6862 Hayenhurst Avenue
Van Nuys, CA 91406
(818) 787-7380
(818) 787-4275 (Fax)
[Multi-adsorbent trap/dry purge]

Graseby
500 Technology Ct.
Smyrna, Georgia 30082
(770) 319-9999
(770) 319-0336 (Fax)
(800) 241-6898
[Controlled Desorption Trap]

Varian Chromatography System
2700 Mitchell Drive
Walnut Creek, California 94898
(510) 945-2196
(510) 945-2335 (FAX)
[Variable Temperature Adsorption Trap]

APPENDIX B.

COMMENT ON CANISTER CLEANING PROCEDURES

The canister cleaning procedures given in Section 8.4 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 can be relaxed. However, the ultimate vacuum achieved during cleaning should always be <0.2mm Hg.

Canister cleaning as described in Section 8.4 and illustrated in Figure 10 requires components with special features. The vacuum gauge shown in Figure 10 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 being 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 typically more exacting than the current certification requirement 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.

APPENDIX C.

LISTING OF COMMERCIAL MANUFACTURERS AND RE-SUPPLIERS OF SPECIALLY-PREPARED CANISTERS

BRC/Rasmussen
17010 NW Skyline Blvd.
Portland. Oregon 97321
(503) 621-1435

Meriter
1790 Potrero Drive
San Jose. CA 95124
(408) 265-6482

Restek Corporation
110 Benner Circle
Bellefonte. PA 16823-8812
(814) 353-1300
(800) 356-1688

Scientific Instrumentation Specialists
P.O. Box 8941
815 Courtney Street
Moscow. ID 83843
(208) 882-3860

Graseby
500 Technology Ct.
Smyrna. Georgia 30082
(404) 319-9999
(800) 241-6898

XonTech Inc.
6862 Hayenhurst Avenue
Van Nuys. CA 91406
(818) 787-7380

APPENDIX D.

LISTING OF COMMERCIAL SUPPLIERS OF PERMEATION TUBES AND SYSTEMS

Kin-Tek
504 Laurel St.
Lamarque, Texas 77568
(409) 938-3627
(800) 326-3627

Vici Metronics, Inc.
2991 Corvin Drive
Santa Clara, CA 95051
(408) 737-0550

Analytical Instrument Development, Inc.
Rt. 41 and Newark Rd.
Avondale, PA 19311
(215) 268-3181

Ecology Board, Inc.
9257 Independence Ave.
Chatsworth, CA 91311
(213) 882-6795

Tracor, Inc.
6500 Tracor Land
Austin, TX
(512) 926-2800

Metronics Associates, Inc.
3201 Porter Drive
Standford Industrial Park
Palo Alto, CA 94304
(415) 493-5632

TABLE 1. VOLATILE ORGANIC COMPOUNDS ON THE TITLE III CLEAN AIR AMENDMENT LIST-- MEMBERSHIP IN COMPENDIUM METHOD TO-14A LIST AND THE SOW-CLP LIST OF VOCs

Compound	CAS No.	BP (°C)	v.p. (mmHg)^1	MW^1	TO-14A	CLP-SOW
Methyl chloride (chloromethane); CH3Cl	74-87-3	-23.7	3.8 x 10	50.5	X	X
Carbonyl sulfide; COS	463-58-1	-50.0	3.7 x 10	60.1		
Vinyl chloride (chloroethene); C2H3Cl	75-01-4	-14.0	3.2 x 10	62.5	X	X
Diazomethane; CH2N2	334-88-3	-23.0	2.8 x 10	42.1		
Formaldehyde; CH2O	50-00-0	-19.5	2.7 x 10	30		
1,3-Butadiene; C4H6	106-99-0	-4.5	2.0 x 10	54		X
Methyl bromide (bromomethane); CH3Br	74-83-9	3.6	1.8 x 10	94.9	X	X
Phosgene; CCl2O	75-44-5	8.2	1.2 x 10	99		
Vinyl bromide (bromoethene); C2H3Br	593-60-2	15.8	1.1 x 10	107		
Ethylene oxide; C2H4O	75-21-8	10.7	1.1 x 10	44		
Ethyl chloride (chloroethane); C2H5Cl	75-00-3	12.5	1.0 x 10	64.5	X	X
Acetaldehyde (ethanal); C2H4O	75-07-0	21.0	952	44		
Vinylidene chloride (1,1-dichloroethylene); C2H2Cl2	75-35-4	31.7	500	97	X	X
Propylene oxide; C3H6O	75-56-9	34.2	445	58		
Methyl iodide (iodomethane); CH3I	74-88-4	42.4	400	141.9		
Methylene chloride; CH2Cl2	75-09-2	40.0	349	84.9	X	X
Methyl isocyanate; C2H3NO	624-83-9	59.6	348	57.1		
Allyl chloride (3-chloropropene); C3H5Cl	107-05-1	44.5	340	76.5	X	X
Carbon disulfide; CS2	75-15-0	46.5	260	76		
Methyl tert-butyl ether; C5H12O	1634-04-4	55.2	249	86		
Propionaldehyde; C2H5CHO	123-38-6	49.0	235	58.1		
Ethylidene dichloride (1,1-dichloroethane); C2H4Cl2	75-34-3	57.0	230	99	X	

TABLE 1. (continued)

Compound	CAS No.	BP (°C)	v.p. (mmHg)^1	MW^1	TO-14A	CLP-SOW
Chloroprene (2-chloro-1,3-butadiene); C4H5Cl	126-99-8	59.4	226	88.5		
Chloromethyl methyl ether; C2H5ClO	107-30-2	59.0	224	80.5		
Acrolein (2-propenal); C3H4O	107-02-8	52.5	220	56		X
1,2-Epoxybutane (1,2-butylene oxide); C4H8O	106-88-7	63.0	163	72		
Chloroform; CHCl3	67-66-3	61.2	160	119	X	X
Ethyleneimine (aziridine); C2H5N	151-56-4	56	160.0	43		
1,1-Dimethylhydrazine; C2H8N2	57-14-7	63	157.0	60.0		
Hexane; C6H14	110-54-3	69.0	120	86.2	X	
1,2-Propyleneimine (2-methylaziridine); C3H7N	75-55-8	66.0	112	57.1		
Acrylonitrile (2-propenenitrile); C3H3N	107-13-1	77.3	100	53	X	
Methyl chloroform (1,1,1-trichloroethane); C2H3Cl3	71-55-6	74.1	100	133.4	X	X
Methanol; CH4O	67-56-1	65.0	92.0	32		X
Carbon tetrachloride; CCl4	56-23-5	76.7	90.0	153.8	X	X
Vinyl acetate; C4H6O2	108-05-4	72.2	83.0	86		X
Methyl ethyl ketone (2-butanone); C4H8O	78-93-3	79.6	77.5	72		X
Benzene; C6H6	71-43-2	80.1	76.0	78	X	X
Acetonitrile (cyanomethane); C2H3N	75-05-8	82	74.0	41.0		X
Ethylene dichloride (1,2-dichloroethane); C2H4Cl2	107-06-2	83.5	61.5	99	X	X
Triethylamine; C6H15N	121-44-8	89.5	54.0	101.2		
Methylhydrazine; CH6N2	60-34-4	87.8	49.6	46.1		
Propylene dichloride (1,2-dichloropropane); C3H6Cl2	78-87-5	97.0	42.0	113	X	X
2,2,4-Trimethyl pentane C8H18	540-84-1	99.2	40.6	114		
1,4-Dioxane (1,4-Diethylene oxide); C4H8O2	123-91-1	101	37.0	88		
Bis(chloromethyl) ether; C2H4Cl2O	542-88-1	104	30.0	115		
Ethyl acrylate; C5H8O2	140-88-5	100	29.3	100		
Methyl methacrylate; C5H8O2	80-62-6	101	28.0	100.1		

TABLE 1. (continued)

Compound	CAS No.	BP (°C)	v.p. (mmHg)[1]	MW[1]	TO-14A	CLP-SOW
Methyl methacrylate; C5H8O2	80-62-101	101	28.0	100.1		
1,3-Dichloropropene; C3H4Cl2 (cis)	542-75-6	112	27.8	111	X	X
Toluene; C7H8	108-88-3	111	22.0	92	X	X
Trichloroethylene; C2HCl3	79-01-6	87.0	20.0	131.4	X	X
1,1,2-Trichloroethane; C2H3Cl3	79-00-5	114	19.0	133.4	X	X
Tetrachloroethylene; C2Cl4	127-18-4	121	14.0	165.8	X	X
Epichlorohydrin (1-chloro-2,3-epoxy propane); C3H5ClO	106-89-8	117	12.0	92.5		
Ethylene dibromide (1,2-dibromoethane); C2H4Br2	106-93-4	132	11.0	187.9	X	X
N-Nitroso-N-methylurea; C2H5N3O2	684-93-5	124	10.0	103		
2-Nitropropane; C3H7NO2	79-46-9	120	10.0	89		
Chlorobenzene; C6H5Cl	108-90-7	132	8.8	112.6	X	X
Ethylbenzene; C8H10	100-41-4	136	7.0	106	X	X
Xylenes (isomer & mixtures); C8H10	1330-20-7	142	6.7	106.2	X	X
Styrene; C8H8	100-42-5	145	6.6	104	X	X
p-Xylene; C8H10	106-42-3	138	6.5	106.2	X	X
m-Xylene; C8H10	108-38-3	139	6.0	106.2	X	X
Methyl isobutyl ketone (hexone); C6H12O	108-10-1	117	6.0	100.2		
Bromoform (tribromomethane); CHBr3	75-25-2	149	5.6	252.8		
1,1,2,2-Tetrachloroethane; C2H2Cl4	79-34-5	146	5.0	167.9	X	X
o-Xylene; C8H10	95-47-6	144	5.0	106.2	X	X
Dimethylcarbamyl chloride; C3H6ClNO	79-44-7	166	4.9	107.6		
N-Nitrosodimethylamine; C2H6N2O	62-75-9	152	3.7	74		
Beta-Propiolactone; C3H4O2	57-57-8	Decomposes at 162	3.4	72		
Cumene (isopropylbenzene); C9H12	98-82-8	153	3.2	120		

TABLE 1. (continued)

Compound	CAS No.	BP (°C)	v.p. (mmHg)[1]	MW[1]	TO-14A	CLP-SOW
Cumene (isopropylbenzene); C9H12	98-82-8	153	3.2	120		
Acrylic acid; C3H4O2	79-10-7	141	3.2	72		
N,N-Dimethylformamide; C3H7NO	68-12-2	153	2.7	73		
1,3-Propane sultone; C3H6O3S	1120-71-4	180/30mm	2.0	122.1		
Acetophenone; C8H8O	98-86-2	202	1.0	120		
Dimethyl sulfate; C2H6O4S	77-78-1	188	1.0	126.1		
Benzyl chloride (a-chlorotoluene); C7H7Cl	100-44-7	179	1.0	126.6	X	X
1,2-Dibromo-3-chloropropane; C3H5Br2Cl	96-12-8	196	0.80	236.4		
Bis(2-Chloroethyl)ether; C4H8Cl2O	111-44-4	178	0.71	143		
Chloroacetic acid; C2H3ClO2	79-11-8	189	0.69	94.5		
Aniline (aminobenzene); C6H7N	62-53-3	184	0.67	93		
1,4-Dichlorobenzene (p-); C6H4Cl2	106-46-7	173	0.60	147	X	X
Ethyl carbamate (urethane); C3H7NO2	51-79-6	183	0.54	89		
Acrylamide; C3H5NO	79-06-1	125/25 mm	0.53	71		
N,N-Dimethylaniline; C8H11N	121-69-7	192	0.50	121		
Hexachloroethane; C2Cl6	67-72-1	Sublimes at 186	0.40	236.7		
Hexachlorobutadiene; C4Cl6	87-68-3	215	0.40	260.8	X	X
Isophorone; C9H14O	78-59-1	215	0.38	138.2		
N-Nitrosomorpholine; C4H8N2O2	59-89-2	225	0.32	116.1		
Styrene oxide; C8H8O	96-09-3	194	0.30	120.2		
Diethyl sulfate; C4H10O4S	64-67-5	208	0.29	154		
Cresylic acid (cresol isomer mixture);C7H8O	1319-77-3	202	0.26	108		
o-Cresol; C7H8O	95-48-7	191	0.24	108		
Catechol (o-hydroxyphenol); C6H6O2	120-80-9	240	0.22	110		
Phenol; C6H6O	108-95-2	182	0.20	94		

TABLE 1. (continued)

Compound	CAS No.	BP (°C)	v.p. (mmHg)[1]	MW[1]	TO-14A	CLP-SOW
Catechol (o-hydroxyphenol); C6H6O2	120-80-9	240	0.22	110		
Phenol; C6H6O	108-95-2	182	0.20	94		
1,2,4-Trichlorobenzene; C6H3Cl3	120-82-1	213	0.18	181.5	X	X
nitrobenzene C6H5NO2	98-95-3	211	0.15	123		

[1] Vapor pressure (v.p.), boiling point (BP) and molecularweight (MW) data from:
(a)D. L. Jones and J. bursey, "Simultaneous Control of PM-10 and Hazardous Air Pollutants II: Rationale for Selection of Hazardous Air Pollutants as Potential Particulate Matter," Report EPA-452/R-93/013, U. S. Environmental Protection Agency, Research Triangle Park, NC. October 1992;
(b)R. C. Weber, P. A. Parker, and M. Bowser. Vapor Pressure Distribution of Selected Organic Chemicals, Report EPA-600/2-81-021, U. S. Environmental Protection Agency, Cincinnati, OH, February 1981; and
(c)R. C. Weast, ed., "CRC Handbook of Chemistry and Physics," 59th edition, CRC Press, Boca Raton, 1979.

TABLE 2. CHARACTERISTIC MASSES (M/Z) USED FOR QUANTIFYING THE TITLE III CLEAN AIR ACT AMENDMENT COMPOUNDS

Compound	CAS No.	Primary Ion	Secondary Ion
Methyl chloride (chloromethane): CH3Cl	74-87-3	50	52
Carbonyl sulfide: COS	463-58-1	60	62
Vinyl chloride (chloroethene): C2H3Cl	75-01-4	62	64
Diazomethane: CH2N2	334-88-3	42	41
Formaldehyde: CH2O	50-00-0	29	30
1,3-Butadiene: C4H6	106-99-0	39	54
Methyl bromide (bromomethane): CH3Br	74-83-9	94	96
Phosgene: CCl2O	75-44-5	63	65
Vinyl bromide (bromoethene): C2H3Br	593-60-2	106	108
Ethylene oxide: C2H4O	75-21-8	29	44
Ethyl chloride (chloroethane): C2H5Cl	75-00-3	64	66
Acetaldehyde (ethanal): C2H4O	75-07-0	44	29, 43
Vinylidene chloride (1,1-dichloroethylene): C2H2Cl2	75-35-4	61	96
Propylene oxide: C3H6O	75-56-9	58	57
Methyl iodide (iodomethane): CH3I	74-88-4	142	127
Methylene chloride: CH2Cl2	75-09-2	49	84, 86
Methyl isocyanate: C2H3NO	624-83-9	57	56
Allyl chloride (3-chloropropene): C3H5Cl	107-05-1	76	41, 78
Carbon disulfide: CS2	75-15-0	76	44, 78
Methyl tert-butyl ether: C5H12O	1634-04-4	73	41, 53
Propionaldehyde: C2H5CHO	123-38-6	58	29, 57
Ethylidene dichloride (1,1-dichloroethane): C2H4Cl2	75-34-3	63	65, 27
Chloroprene (2-chloro-1,3-butadiene): C4H5Cl	126-99-8	88	53, 90
Chloromethyl methyl ether: C2H5ClO	107-30-2	45	29, 49
Acrolein (2-propenal): C3H4O	107-02-8	56	55
1,2-Epoxybutane (1,2-butylene oxide): C4H8O	106-88-7	42	41, 72
Chloroform: CHCl3	67-66-3	83	85, 47
Ethyleneimine (aziridine): C2H5N	151-56-4	42	43
1,1-Dimethylhydrazine: C2H8N2	57-14-7	60	45, 59
Hexane: C6H14	110-54-3	57	41, 43
1,2-Propyleneimine (2-methylazindine): C3H7N	75-55-8	56	57, 42
Acrylonitrile (2-propenenitrile): C3H3N	107-13-1	53	52
Methyl chloroform (1,1,1 trichloroethane): C2H3Cl3	71-55-6	97	99, 61
Methanol: CH4O	67-56-1	31	29
Carbon tetrachloride: CCl4	56-23-5	117	119
Vinyl acetate: C4H6O2	108-05-4	43	86
Methyl ethyl ketone (2-butanone): C4H8O	78-93-3	43	72

TABLE 2. (continued)

Compound	CAS No.	Primary Ion	Secondary Ion
Benzene: C6H6	71-43-2	78	77.50
Acetonitrile (cyanomethane): C2H3N	75-05-8	41	40
Ethylene dichloride (1,2-dichloroethane): C2H4Cl2	107-06-2	62	64. 27
Triethylamine: C6H15N	121-44-8	86	58. 101
Methylhydrazine: CH6N2	60-34-4	46	31. 45
Propylene dichloride (1,2-dichloropropane): C3H6Cl2	78-87-5	63	41. 62
2,2,4-Trimethyl pentane: C8H18	540-84-1	57	41. 56
1,4-Dioxane (1,4 Diethylene oxide): C4H8O2	123-91-1	88	58
Bis(chloromethyl) ether: C2H4Cl2O	542-88-1	79	49. 81
Ethyl acrylate: C5H8O2	140-88-5	55	73
Methyl methacrylate: C5H8O2	80-62-6	41	69. 100
1,3-Dichloropropene: C3H4Cl2 (cis)	542-75-6	75	39. 77
Toluene: C7H8	108-88-3	91	92
Trichloethylene: C2HCl3	79-01-6	130	132. 95
1,1,2-Trichloroethane: C2H3Cl3	79-00-5	97	83. 61
Tetrachloroethylene: C2Cl4	127-18-4	166	164. 131
Epichlorohydrin (l-chloro-2,3-epoxy propane): C3H5ClO	106-89-8	57	49. 62
Ethylene dibromide (1,2-dibromoethane): C2H4Br2	106-93-4	107	109
N-Nitrso-N-methylurea: C2H5N3O2	684-93-5	60	44. 103
2-Nitropropane: C3H7NO2	79-46-9	43	41
Chlorobenzene: C6H5Cl	108-90-7	112	77. 114
Ethylbenzene: C8H10	100-41-4	91	106
Xylenes (isomer & mixtures): C8H10	1330-20-7	91	106
Styrene: C8H8	100-42-5	104	78. 103
p-Xylene: C8H10	106-42-3	91	106
m-Xylene: C8H10	108-38-3	91	106
Methyl isobutyl ketone (hexone): C6H12O	108-10-1	43	58. 100
Bromoform (tribromomethane): CHBr3	75-25-2	173	171. 175
1,1,2,2-Tetrachloroethane: C2H2Cl4	79-34-5	83	85
o-Xylene: C8H10	95-47-6	91	106
Dimethylcarbamyl chloride: C3H6ClNO	79-44-7	72	107
N-Nitrosodimethylamine: C2H6N2O	62-75-9	74	42
Beta-Propiolactone: C3H4O2	57-57-8	42	43
Cumene (isopropylbenzene): C9H12	98-82-8	105	120
Acrylic acid: C3H4O2	79-10-7	72	45. 55
N,N-Dimethylformamide: C3H7NO	68-12-2	73	42. 44
1,3-Propane sultone: C3H6O3S	1120-71-4	58	65. 122

TABLE 2. (continued)

Compound	CAS No.	Primary Ion	Secondary Ion
Acetophenone: C_8H_8O	98-86-2	105	77,120
Dimethyl sulfate: $C_2H_6O_4S$	77-78-1	95	66,96
Benzyl chloride (a-chlorotoluene): C_7H_7Cl	100-44-7	91	126
1,2-Dibromo-3-chloropropane: $C_3H_5Br_2Cl$	96-12-8	57	155, 157
Bis(2-Chloroethyl)ether: $C_4H_8Cl_2O$	111-44-4	93	63, 95
Chloroacetic acid: $C_2H_3ClO_2$	79-11-8	50	45, 60
Aniline (aminobenzene): C_6H_7N	62-53-3	93	66
1,4-Dichlorobenzene (p-): $C_6H_4Cl_2$	106-46-7	146	148, 111
Ethyl carbamate (urethane): $C_3H_7NO_2$	51-79-6	31	44, 62
Acrylamide: C_3H_5NO	79-06-1	44	55, 71
N,N-Dimethylaniline: $C_8H_{11}N$	121-69-7	120	77, 121
Hexachloroethane: C_2Cl_6	67-72-1	201	199, 203
Hexachlorobutadiene: C_4Cl_6	87-68-3	225	227, 223
Isophorone: $C_9H_{14}O$	78-59-1	82	138
N-Nitrosomorpholine: $C_4H_8N_2O_2$	59-89-2	56	86, 116
Styrene oxide: C_8H_8O	96-09-3	91	120
Diethyl sulfate: $C_4H_{10}O_4S$	64-67-5	45	59, 139
Cresylic acid (cresol isomer mixture): C_7H_8O	1319-77-3		
o-Cresol: C_7H_8O	95-48-7	108	107
Catechol (o-hydroxyphenol): $C_6H_6O_2$	120-80-9	110	64
Phenol: C_6H_6O	108-95-2	94	66
1,2,4-Trichlorobenzene: $C_6H_3Cl_3$	120-82-1	180	182, 184
Nitrobenzene: $C_6H_5NO_2$	98-95-3	77	51, 123

TABLE 3. REQUIRED BFB KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria[1]
50	8.0 to 40.0 Percent of m/e 95
75	30.0 to 66.0 Percent of m/e 95
95	Base Peak. 100 Percent Relative Abundance
96	5.0 to 9.0 Percent of m/e 95 (See note)
173	Less than 2.0 Percent of m/e 174
174	50.0 to 120.0 Percent of m/e 95
175	4.0 to 9.0 Percent of m/e 174
176	93.0 to 101.0 Percent of m/e 174
177	5.0 to 9.0 Percent of m/e 176

[1] All ion abundances must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120 percent that of m/z 95.

TABLE 4. METHOD DETECTION LIMITS (MDL)[1]

TO-14A List	Lab #1, SCAN	Lab #2, SIM
Benzene	0.34	0.29
Benzyl Chloride	–	–
Carbon tetrachloride	0.42	0.15
Chlorobenzene	0.34	0.02
Chloroform	0.25	0.07
1,3-Dichlorobenzene	0.36	0.07
1,2-Dibromoethane	–	0.05
1,4-Dichlorobenzene	0.70	0.12
1,2-Dichlorobenzene	0.44	–
1,1-Dichloroethane	0.27	0.05
1,2-Dichloroethane	0.24	–
1,1-Dichloroethene	–	0.22
cis-1,2-Dichloroethene	–	0.06
Methylene chloride	1.38	0.84
1,2-Dichloropropane	0.21	–
cis-1,3-Dichloropropene	0.36	–
trans-1,3-Dichloropropene	0.22	–
Ethylbenzene	0.27	0.05
Chloroethane	0.19	–
Trichlorofluoromethane	–	–
1,1,2-Trichloro-1,2,2-trifluoroethane		–
1,2-Dichloro-1,1,2,2-tetrafluoroethane	–	–
Dichlorodifluoromethane	–	–
Hexachlorobutadiene	–	–
Bromomethane	0.53	–
Chloromethane	0.40	–
Styrene	1.64	0.06
1,1,2,2-Tetrachloroethane	0.28	0.09
Tetrachloroethene	0.75	0.10
Toluene	0.99	0.20
1,2,4-Trichlorobenzene	–	–
1,1,1-Trichloroethane	0.62	0.21
1,1,2-Trichloroethane	0.50	–
Trichloroethene	0.45	0.07
1,2,4-Trimethylbenzene	–	–
1,3,5-Trimethylbenzene	–	–
Vinyl Chloride	0.33	0.48
m,p-Xylene	0.76	0.08
o-Xylene	0.57	0.28

[1] Method Detection Limits (MDLs) are defined as the product of the standard deviation of seven replicate analyses and the student's "t" test value for 99% confidence. For Lab #2, the MDLs represent an average over four studies. MDLs are for MS/SCAN for Lab #1 and for MS/SIM for Lab #2.

TABLE 5. SUMMARY OF EPA DATA ON REPLICATE PRECISION (RP) FROM EPA NETWORK OPERATIONS[1]

Monitoring Compound Identification	EPA's Urban Air Toxics Monitoring Program (UATMP)			EPA's Toxics Air Monitoring Stations (TAMS)		
	%RP	#	ppbv	%RP	#	ppbv
Dichlorodifluoromethane	--		--	13.9	47	0.9
Methylene chloride	16.3	07	4.3	19.4	47	0.6
1,2-Dichloroethane	36.2	31	1.6	--	--	--
1,1,1-Trichloroethane	14.1	44	1.0	10.6	47	2.0
Benzene	12.3	56	1.6	4.4	47	1.5
Trichloroethene	12.8	08	1.3	--	--	--
Toluene	14.7	76	3.1	3.4	47	3.1
Tetrachloroethene	36.2	12	0.8	--	--	--
Chlorobenzene	20.3	21	0.9	--	--	--
Ethylbenzene	14.6	32	0.7	5.4	47	0.5
m-Xylene	14.7	75	4.0	5.3	47	1.5
Styrene	22.8	59[2]	1.1	8.7	47	0.2[2]
o-Xylene	--		--	6.0	47	0.5
p-Xylene	--					
1,3-Dichlorobenzene	49.1	06	0.6	--	--	--
1,4-Dichlorobenzene	14.7	14	6.5	--	--	--

[1] Denotes the number of replicate or duplicate analysis used to generate the statistic. The replicate precision is defined as the mean ratio of absolute difference to the average value.

[2] Styrene and o-xylene coelute from the GC column used in UATMP. For the TAMS entries, both values were below detection limits for 18 of 47 replicates and were not included in the calculation.

TABLE 6. AUDIT ACCURACY (AA) VALUES[1] FOR SELECTED COMPENDIUM METHOD TO-14A COMPOUNDS

Selected Compounds From TO-14A List	FY-88 TAMS AA(%), N=30	FY-88 UATMP AA(%), N=3
Vinyl chloride	4.6	17.9
Bromomethane	--	6.4
Trichlorofluoromethane	6.4	--
Methylene chloride	8.6	31.4
Chloroform	--	4.2
1,2-Dichloroethane	6.8	11.4
1,1,1-Trichloroethane	18.6	11.3
Benzene	10.3	10.1
Carbon tetrachloride	12.4	9.4
1,2-Dichloropropane	--	6.2
Trichloroethene	8.8	5.2
Toluene	8.3	12.5
Tetrachloroethene	6.2	--
Chlorobenzene	10.5	11.7
Ethylbenzene	12.4	12.4
o-Xylene	16.2	21.2

[1] Audit accuracy is defined as the relative difference between the audit measurement result and its nominal value divided by the nominal value. N denotes the number of audits averaged to obtain the audit accuracy value. Information is not available for other TO-14A compounds because they were not present in the audit materials.

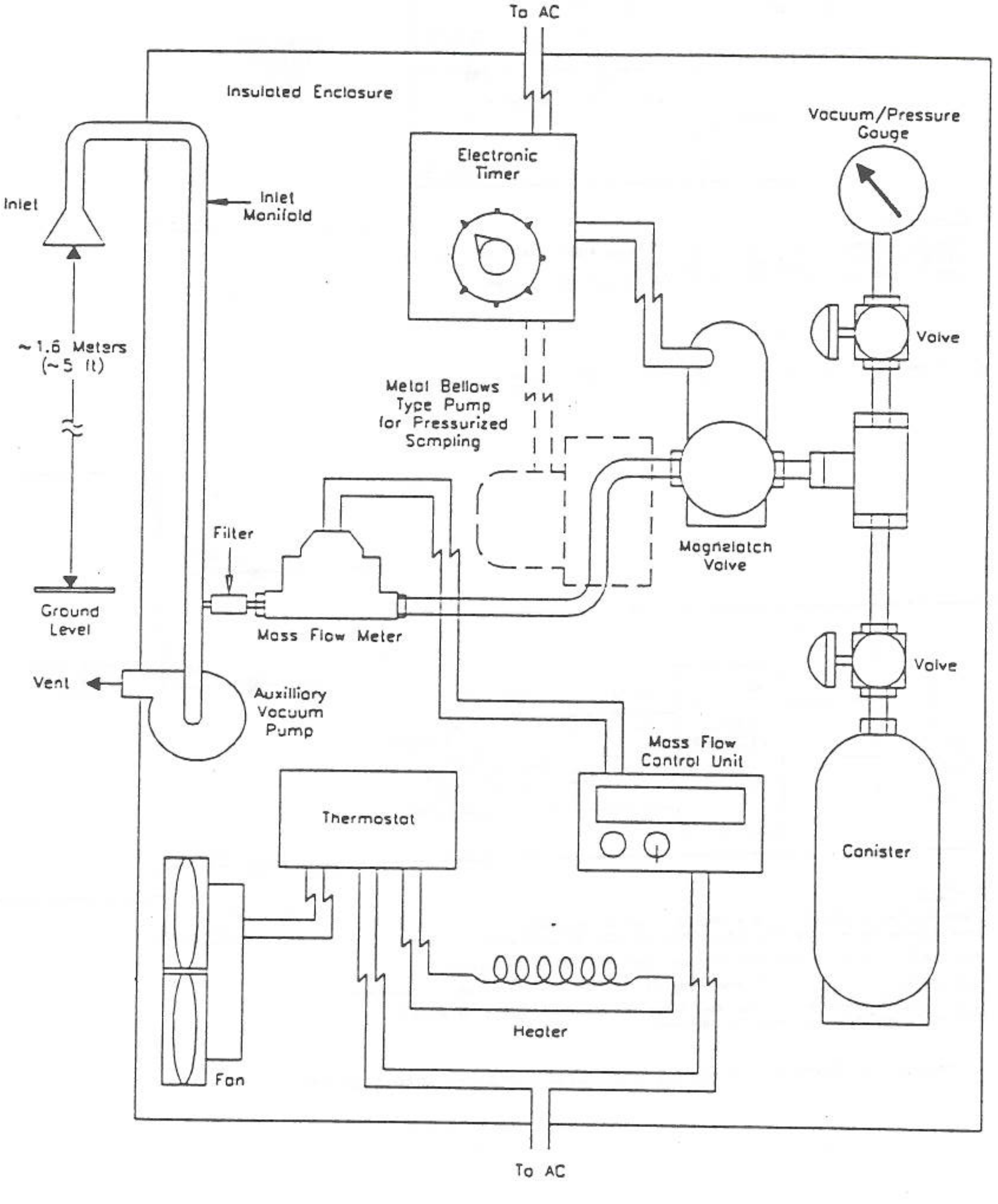
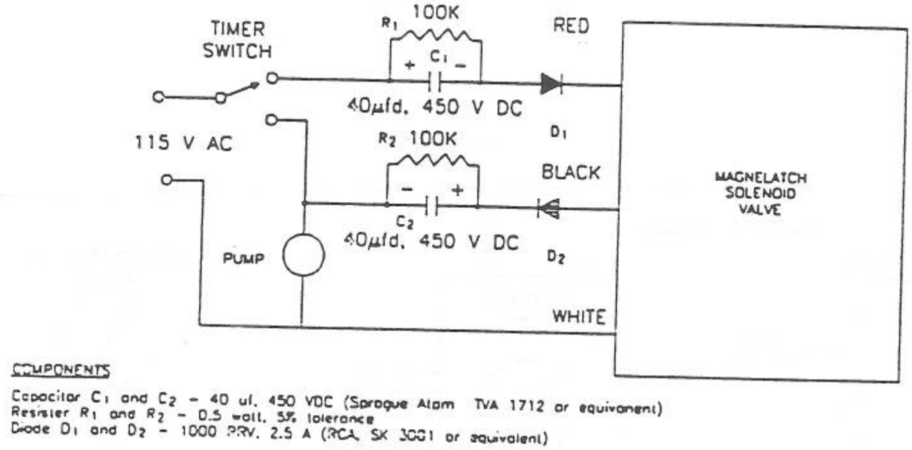


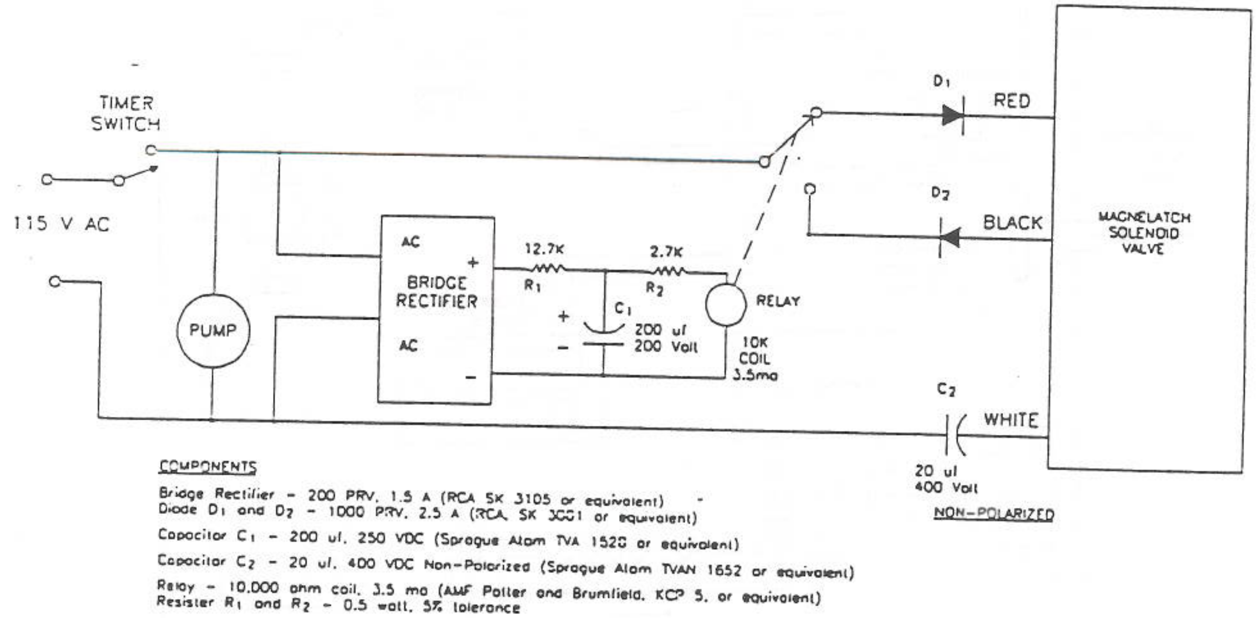
Figure 1. Sampler configuration for subatmospheric pressure or pressurized canister sampling.



COMPONENTS

Capacitor C_1 and C_2 - 40 ul, 450 VDC (Sprague Atom TVA 1712 or equivonent)
Resister R_1 and R_2 - 0.5 watt, 5% tolerance
Diode D_1 and D_2 - 1000 PRV, 2.5 A (RCA, SK 3001 or equivalent)

(a). Simple Circuit for Operating Magnelatch Valve



COMPONENTS

Bridge Rectifier - 200 PRV, 1.5 A (RCA SK 3105 or equivalent)
Diode D_1 and D_2 - 1000 PRV, 2.5 A (RCA, SK 3001 or equivalent)
Capacitor C_1 - 200 ul, 250 VDC (Sprague Atom TVA 1520 or equivalent)
Capacitor C_2 - 20 ul, 400 VDC Non-Polarized (Sprague Atom TVAN 1652 or equivalent)
Relay - 10,000 ohm coil, 3.5 ma (AMF Potter and Brumfield, KCP 5, or equivalent)
Resister R_1 and R_2 - 0.5 watt, 5% tolerance

(b). Improved Circuit Designed to Handle Power Interruptions

Figure 2. Electrical pulse circuits for driving Skinner magnelatch solenoid valve with mechanical timer.

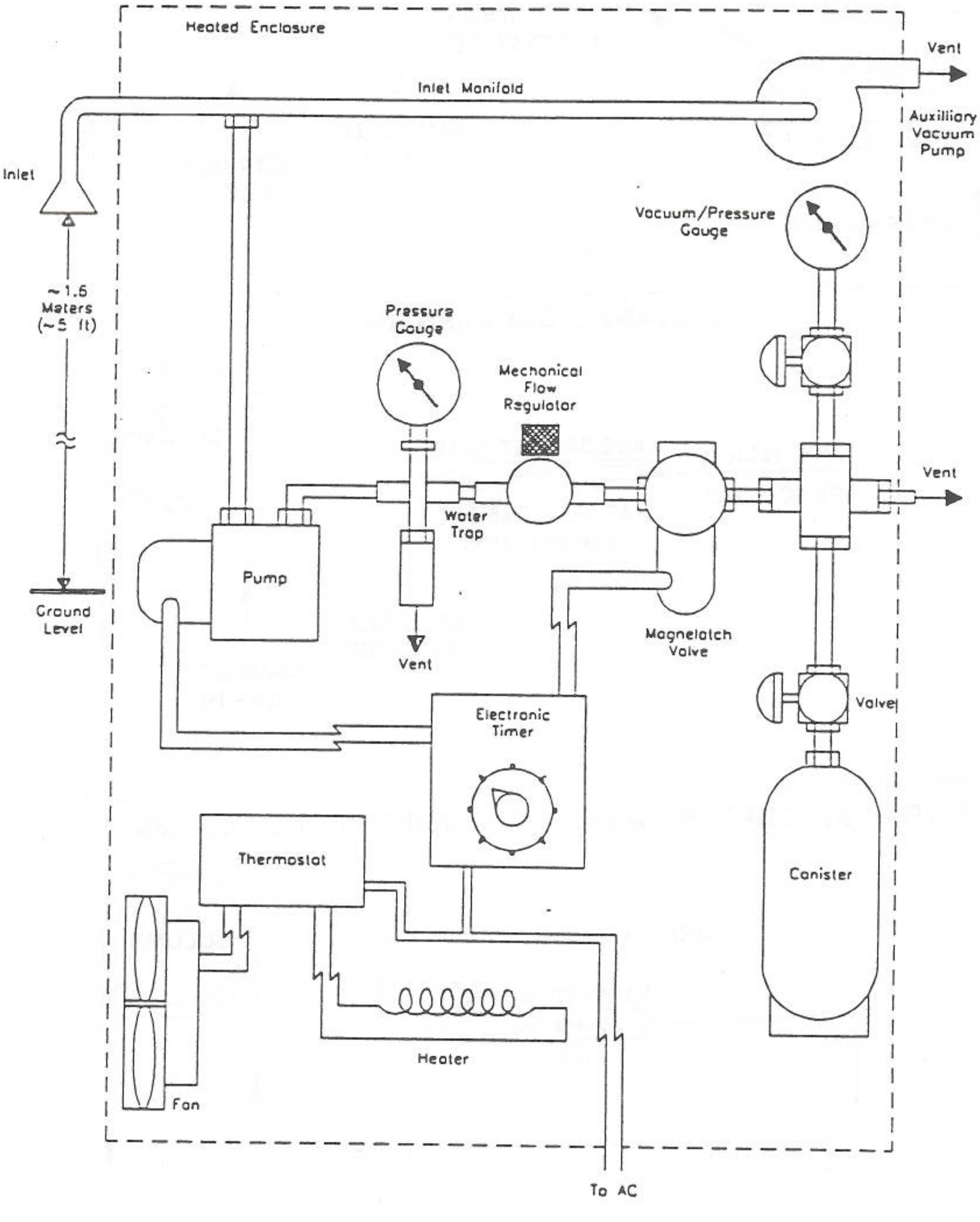
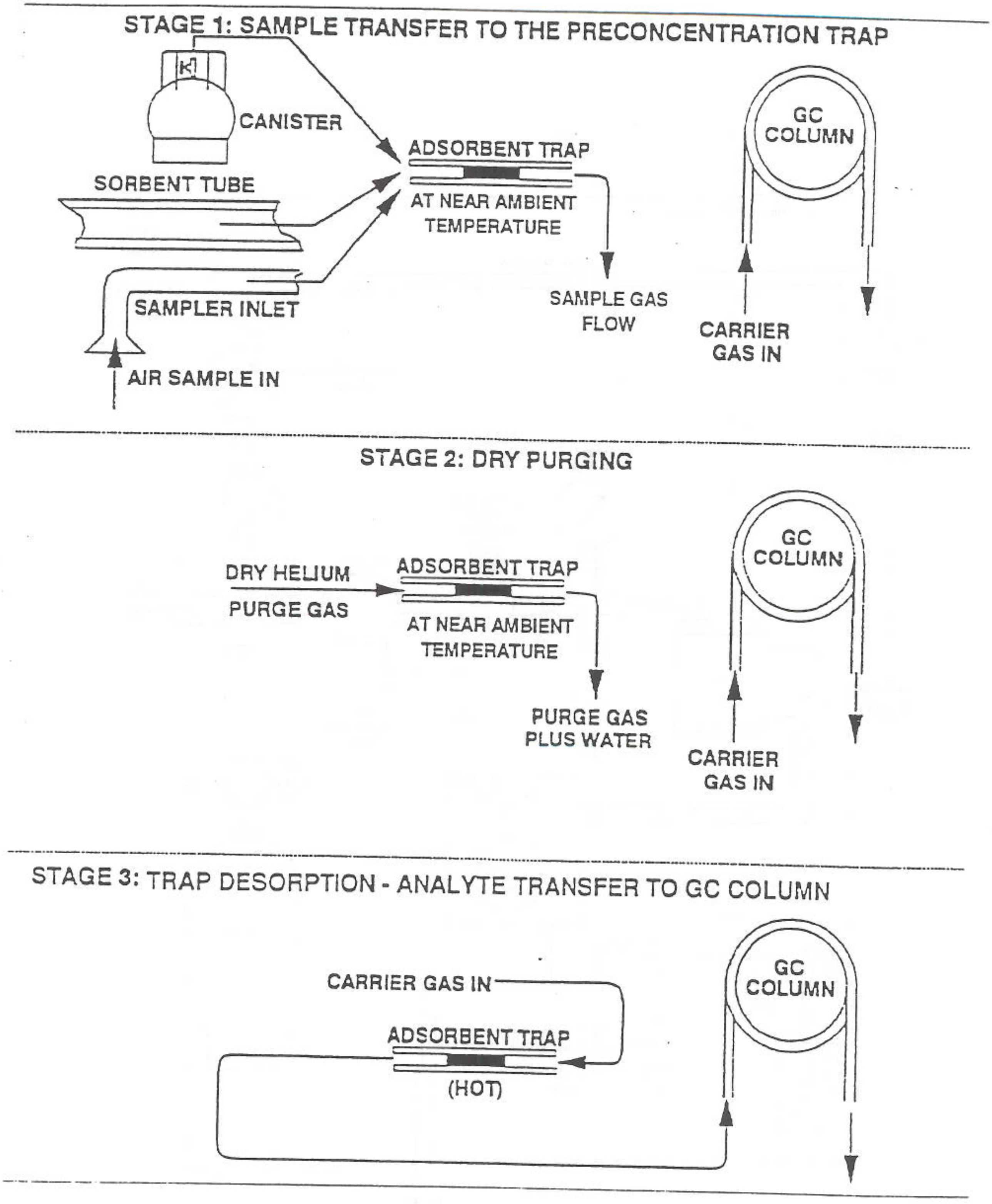


Figure 3. Alternative sampler configuration for pressurized canister sampling.



STAGE 1: SAMPLE TRANSFER TO THE PRECONCENTRATION TRAP

STAGE 2: DRY PURGING

STAGE 3: TRAP DESORPTION - ANALYTE TRANSFER TO GC COLUMN

Figure 4. Illustration of three stages of dry purging of adsorbent trap.

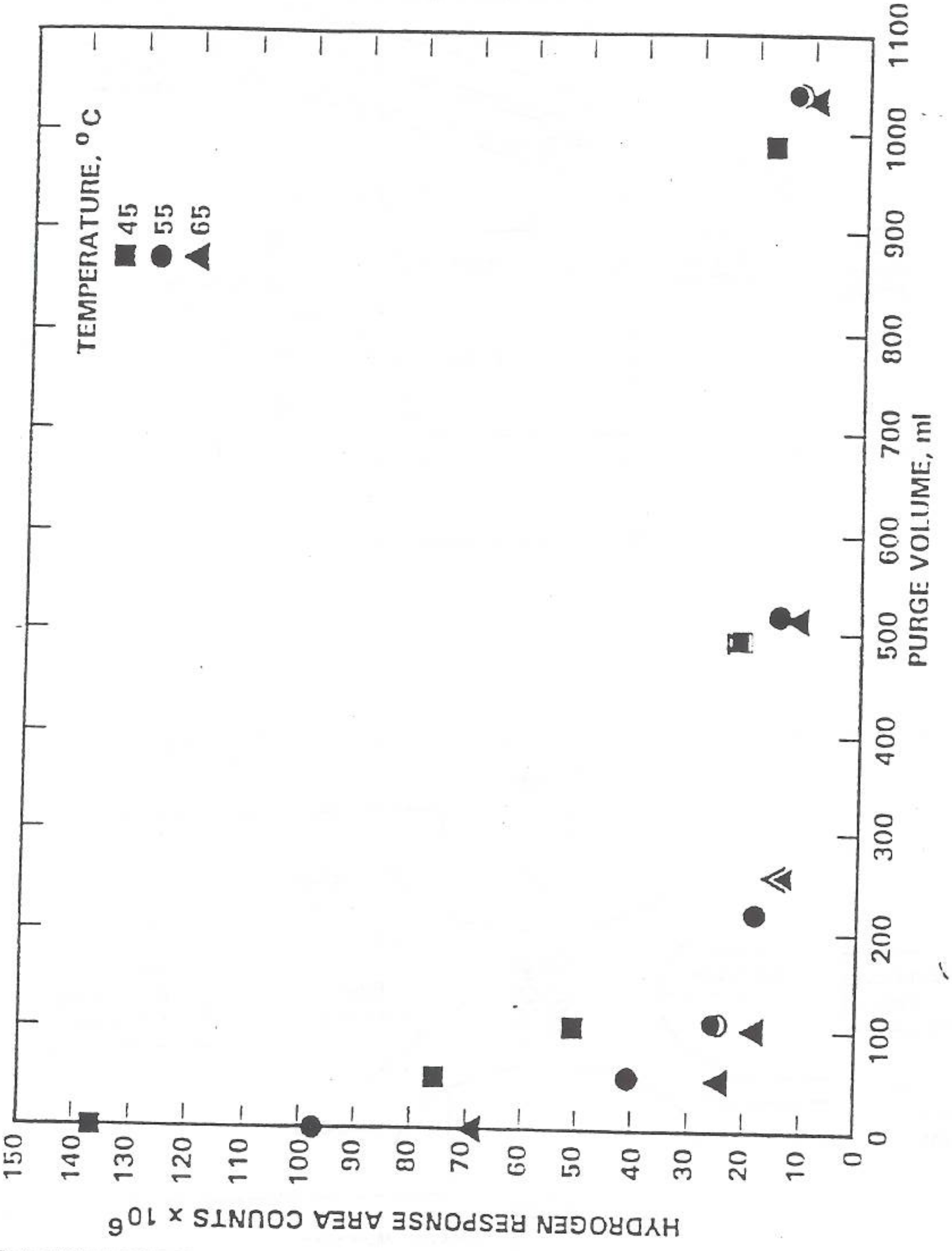


Figure 5. Residual water vapor on VOC concentrator vs. dry He purge volume.

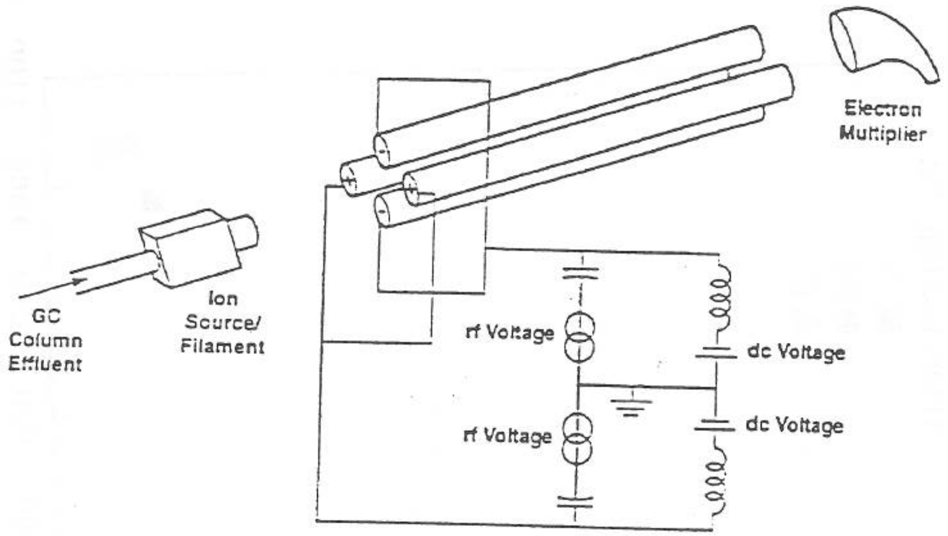


Figure 6. Simplified diagram of a quadrupole mass spectrometer.

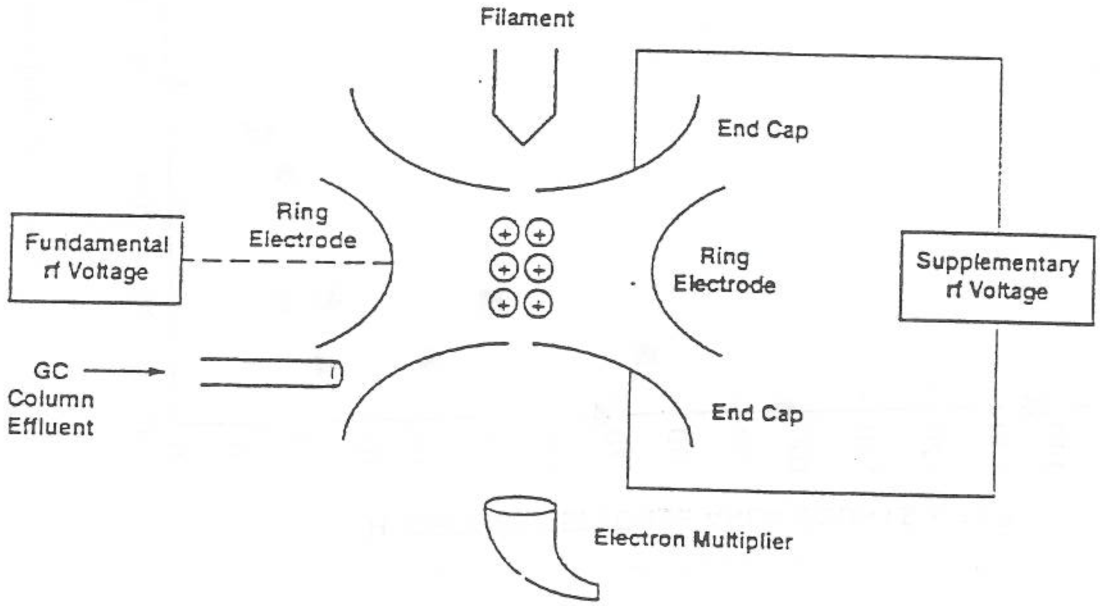


Figure 7. Simplified diagram of an ion trap mass spectrometer.

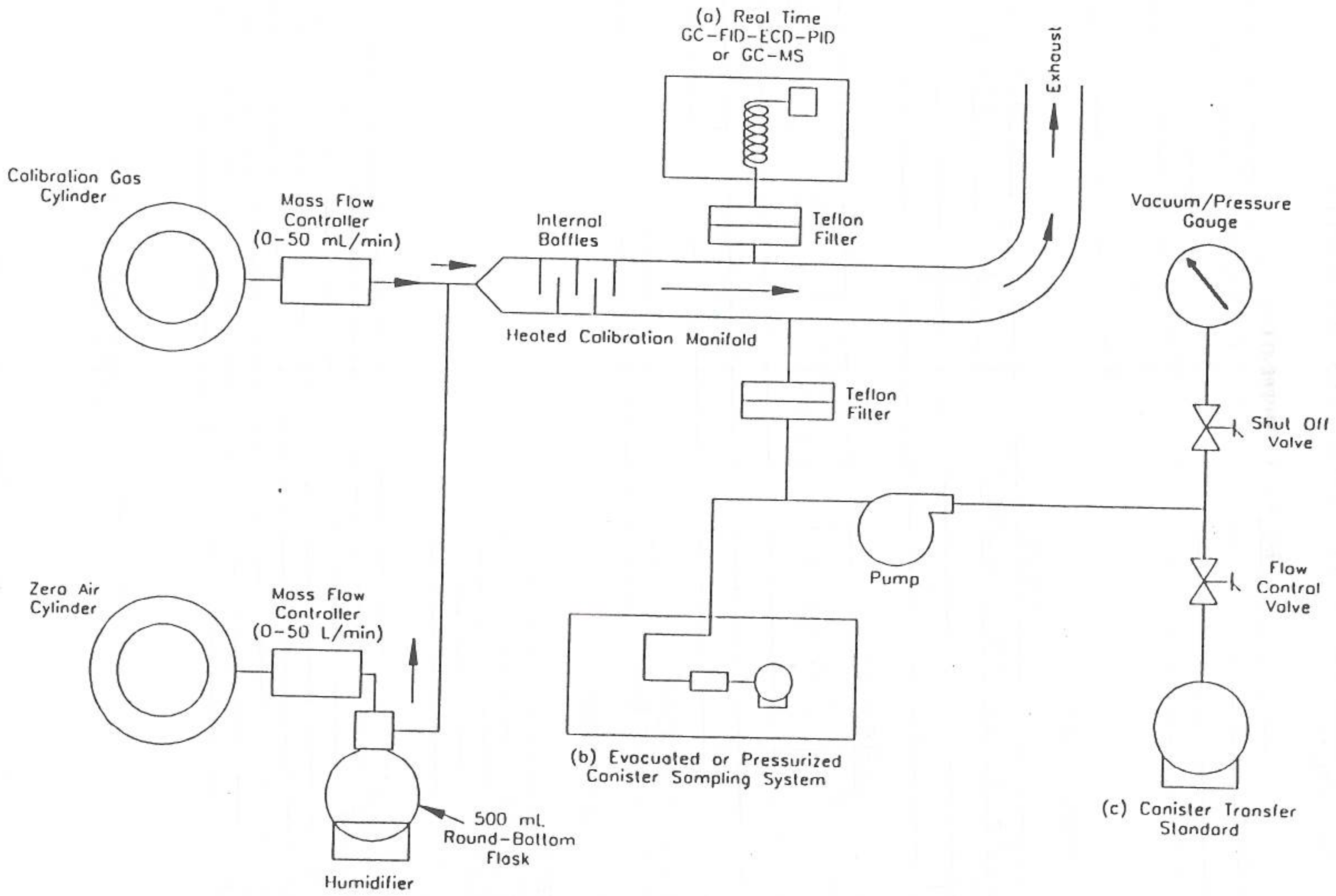


Figure 8. Schematic diagram of calibration system and manifold for
(a) analytical system calibration, (b) testing canister sampling system and (c) preparing canister transfer standards.

COMPENDIUM METHOD TO-15
CANISTER SAMPLING FIELD TEST DATA SHEET

A.GENERAL INFORMATION

SITE LOCATION: ______________________

SITE ADDRESS: ______________________

____

SAMPLING DATE: ______________________

SHIPPING DATE: ______________________

CANISTER SERIAL NO.: ______________________

SAMPLER ID: ______________________

OPERATOR: ______________________

CANISTER LEAK
CHECK DATE: ______________________

B. SAMPLING INFORMATION

	TEMPERATURE			
	INTERIOR	AMBIENT	MAXIMUM	MINIMUM
START				
STOP				

PRESSURE	
CANISTER PRESSURE	

	SAMPLING TIMES	
	LOCAL TIME	ELAPSED TIME METER READING
START		
STOP		

FLOW RATES		
MANIFOLD FLOW RATE	CANISTER FLOW RATE	FLOW CONTROLLER READOUT

SAMPLING SYSTEM CERTIFICATION DATE: ______________________

QUARTERLY RECERTIFICATION DATE: ______________________

C. LABORATORY INFORMATION

DATA RECEIVED: ______________________

RECEIVED BY: ______________________

INITIAL PRESSURE: ______________________

FINAL PRESSURE: ______________________

DILUTION FACTOR: ______________________

ANALYSIS

GC-FID-ECD DATE: ______________________

GC-MSD-SCAN DATE: ______________________

GC-MSD-SIM DATE: ______________________

RESULTS*: ______________________

____

GC-FID-ECD: ______________________

GC-MSD-SCAN: ______________________

GC-MSD-SIM: ______________________

____

SIGNATURE/TITLE

Figure 9. Canister sampling field test data sheet (FTDS).

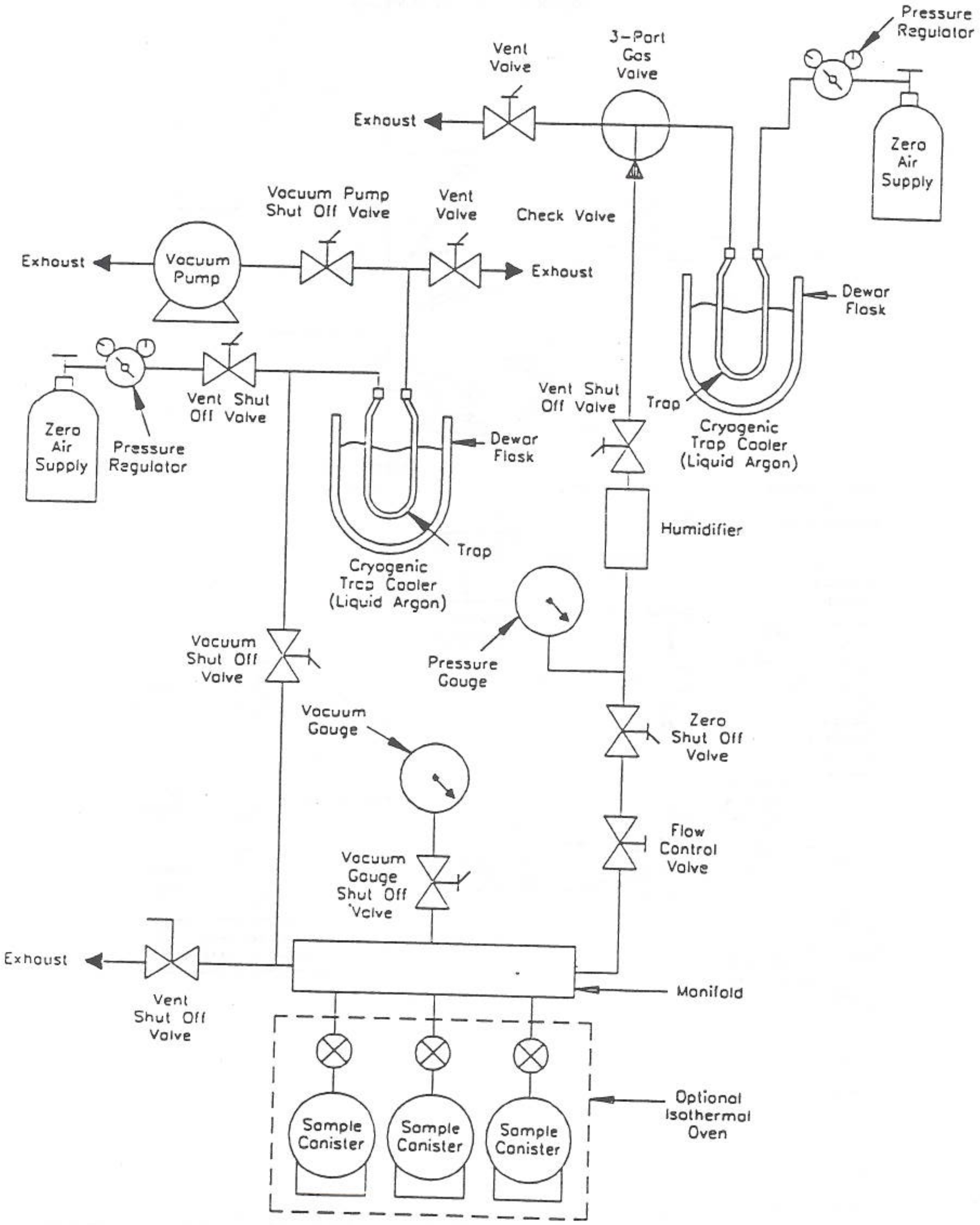


Figure 10. Canister cleaning system.

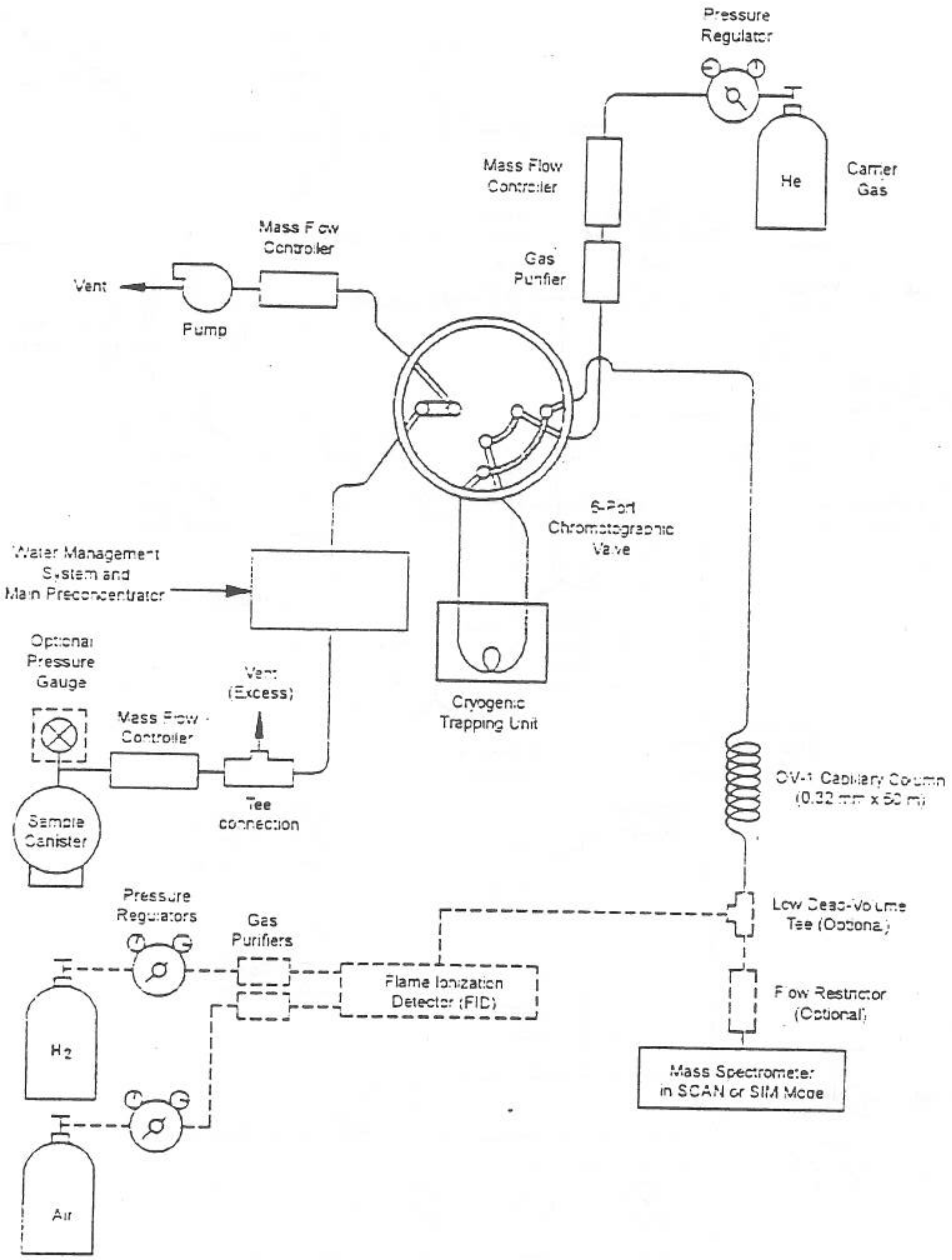
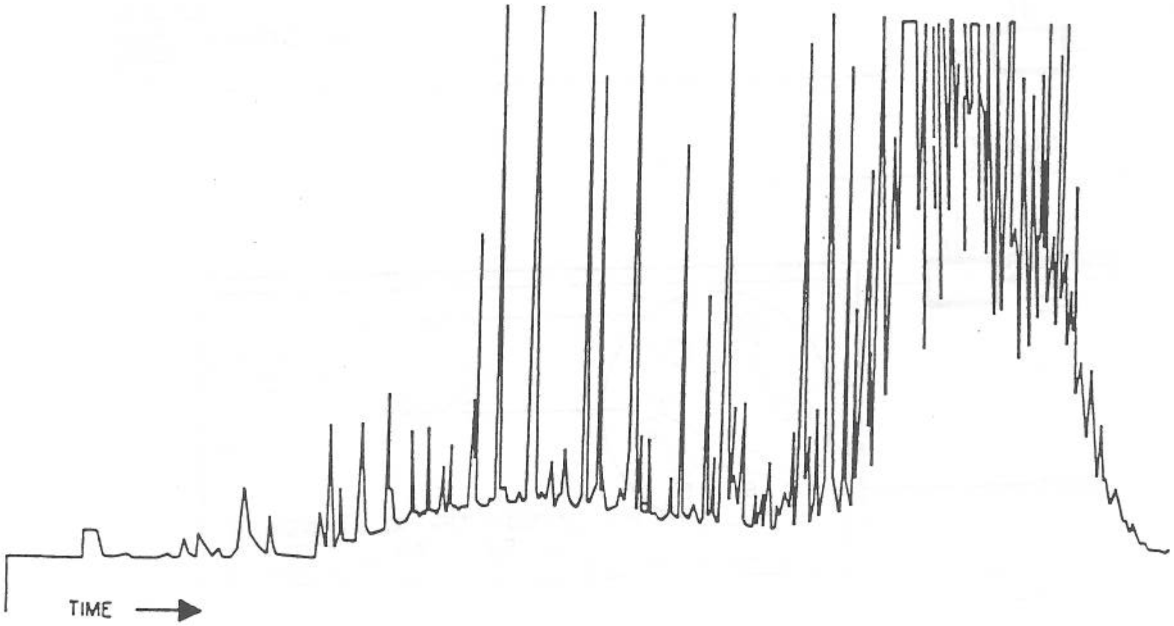


Figure 11. Canister analysis utilizing GC/MS/SCAN/SIM analytical system with optional flame ionization detector with 6-port chromatographic valve in the sample desorption mode.
[Alternative analytical system illustrated in Figure 16.]

(a). Certified Sampler



(b). Contaminated Sampler

Figure 12. Example of humid zero air test results for a clean sample canister (a) and a contaminated sample canister (b).

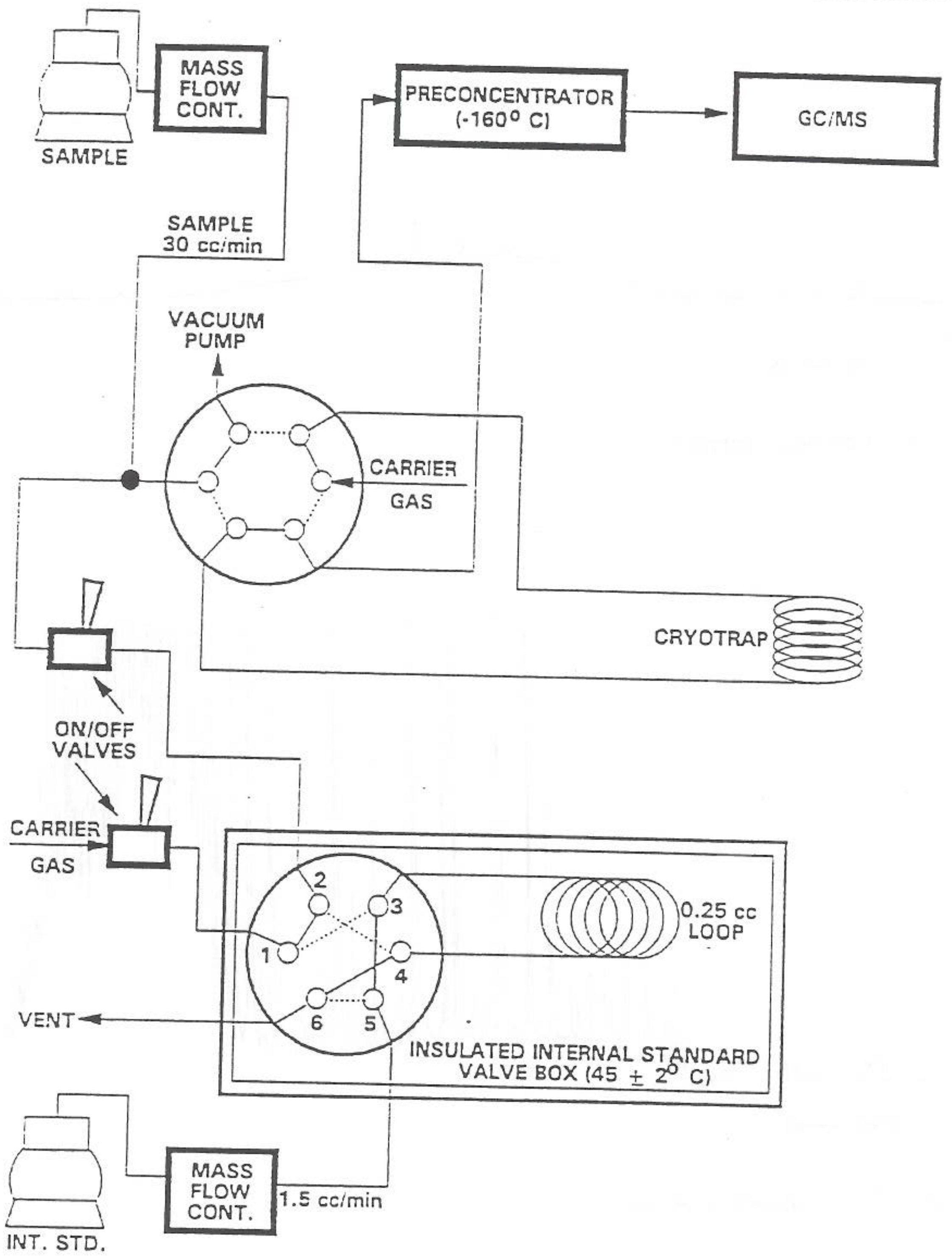


Figure 13. Diagram of design for internal standard addition.

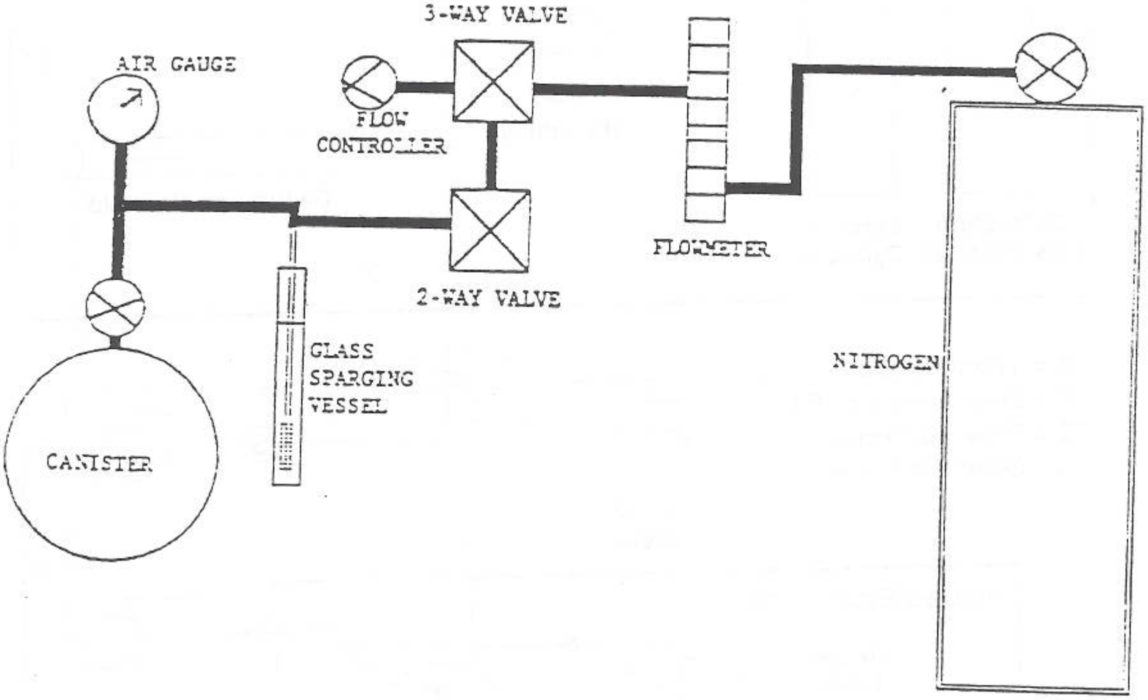


Figure 14. Water method of standard preparation in canisters.

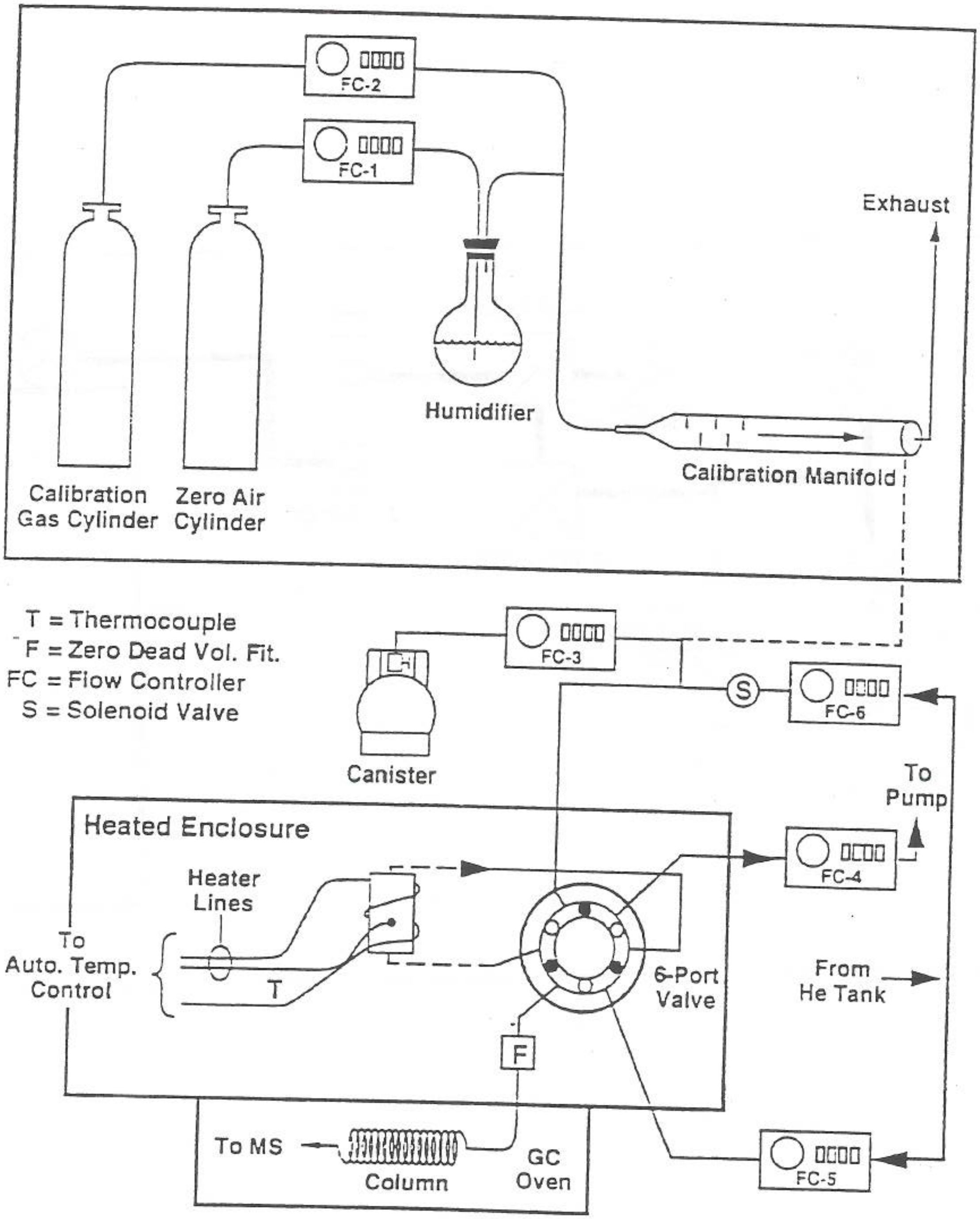


Figure 15. Diagram of the GC/MS analytical system.

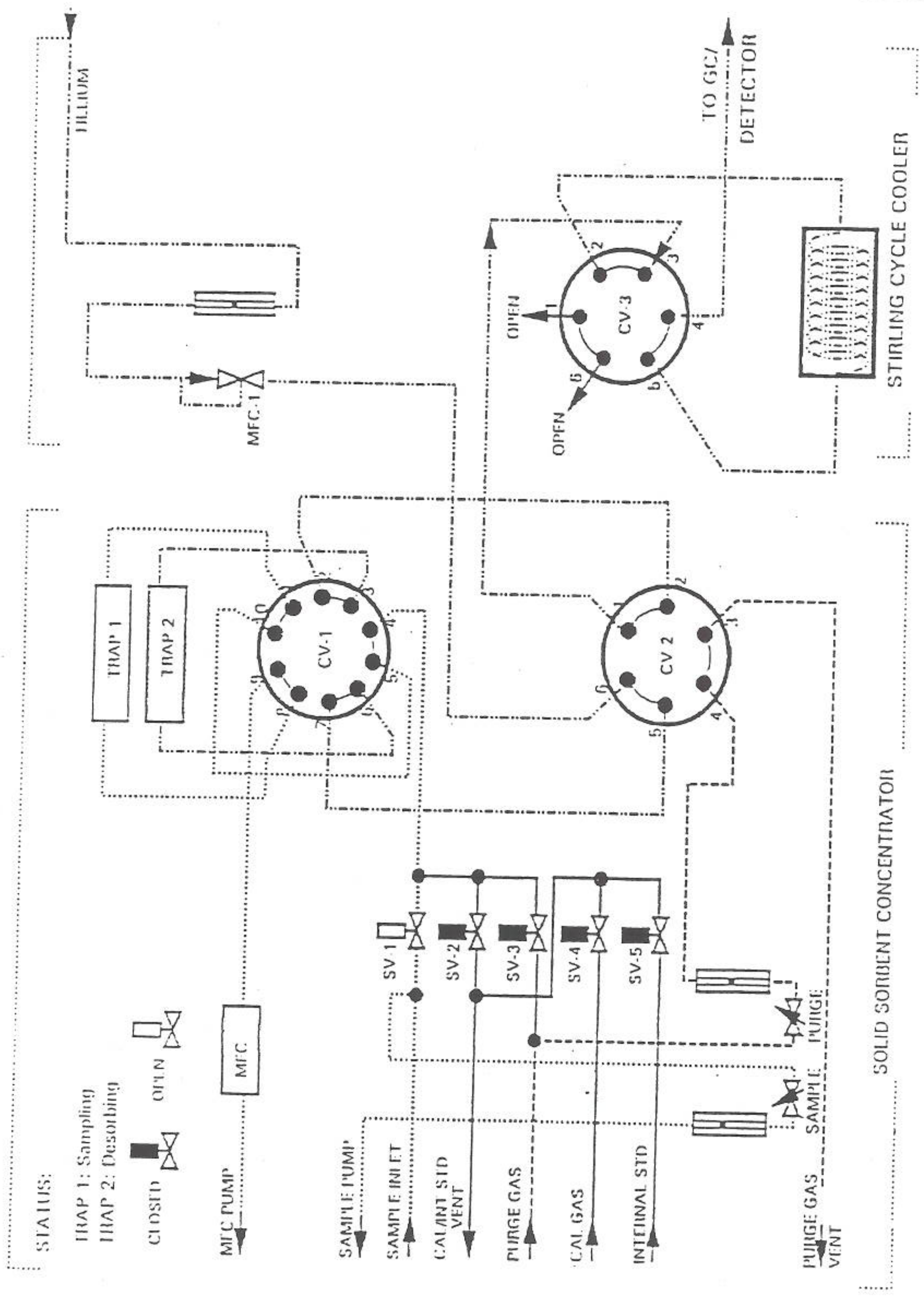


Figure 16. Sample flow diagram of a commercially available concentrator showing the combination of multisorbent tube and cooler (Trap 1 sampling; Trap 2 desorbing).

Attachment A

Pneumatic Head Loss Calculation

Pneumatic Analysis

Project: Jackson Steel - Soil Vapor Extraction - Phase 1 Design Basis

Constants

Density, p =	1.2 kg/m^3 @ 50°F	red = entered value
Absolute viscosity, v =	1.71E-05 kg/m*s	black = calculated value
Kinematic viscosity, k =	1.43E-05 m^2/s	
Gravity, g =	9.81 m/s^2	
Roughness coef., e =	0.0015 mm (drawn tubing - SCH 40 PVC or vacuum flex hose)	

Pipelines	Line 1	Line 2	Line 3	Total	Comments
Flow (cfm)	300	300	300		Line 1 is the well casing.
Flow (m^3/s)	0.1416	0.1416	0.14158		Line 2 is flex hose between well and SVE trailer.
Pipe ID (in)	4	3	3		Line 3 is the SVE Blower discharge.
Pipe ID (m)	0.1016	0.0762	0.0762		
Inlet Area (m^2)	0.00811	0.00456	0.00456		
Velocity (m/s)	17.46	31.05	31.05		
Velocity (ft/s)	57.30	101.86	101.86		
Reynolds #	1.25E+05	1.66E+05	1.66E+05		
e/D	0.000015	0.000020	0.000020		
f, friction factor (dim.)	0.018	0.017	0.034		
Length (ft)	25	60	40	125.0	
Length (m)	7.62	18.29	12.19	38.1	
Minor Losses					
Fittings - Resistance Coefficient (K) values					
Gate Valve	0.18	0.18	0.18		
Ball Valve	0.07	0.07	0.07		
Butterfly Valve					
Elbow, 90°	0.69	0.69	0.69		
Tee, thru	0.46	0.46	0.46		
Tee, branch	1.38	1.38	1.38		
Check, swing	2.3	2.3	2.3		
Expansion	1	1	1		
Contraction	0.5	0.5	0.5		
Fittings - Quantities					
Gate Valve	1		1		
Ball Valve					
Butterfly Valve					
Elbow, 90°		4	4		
Tee, thru		1			
Tee, branch			1		
Check, swing					
Expansion		1	1		
Contraction	1	1	1		
Pressure Loss					
Pipeline losses (m air)	21.0	200.4	267.26	488.7	
Minor losses (m air)	10.6	231.9	285.93	528.4	
Total losses (m air)	31.6	432.3	553.18	1017.1	
Total losses (psi)	0.05	0.74	0.94	1.7	
Total losses (in Hg)	0.11	1.50	1.92	**3.5**	

Total estimated conveyance losses (in Hg) = **3.5**
Estimated dP across two 200 lb carbon vessels in series (in Hg) = **2.9** (or 40 inches H2O)
Estimated maximum required wellhead vacuum (in Hg) = **1.0** (or 13 inches H2O)
Total dP requirements (in Hg) = **7.4**

Note: Pressure loss calculated using Darcy-Weisbach equation; $h_f = f * L/D * V^2/2g + k * V^2/2g$, valid for compressible flow if the calculated pressure drop is less than abou 10% of the inlet pressure (Flow of Fluids Through Valves, Fittings, and Pipe, Technical Paper No. 410, Crane Co., 1988)

where; h_f = pressure loss (meters of air)
f = friction factor (dim., determined using Moody Diagram)
L = length of pipe (m)
D = diameter of pipe (m)
V = fluid velocity (m/s)
g = gravitational constant (m/s^2)

Attachment B
Carbon Modeling Results

CARBONAIR

Temporary Water Treatment Services Carbon • Rental • Service

2731 Nevada Ave N, New Hope, MN 55427 Phone: 800-526-4999 Fax: 763-544-2151 www.carbonair.com

Customer: CH2M Hill
Site: Unknown
Date: 1/5/05

Design Basis:	Flow rate:	300	cfm
	Air temperature:	70	°F (assumed before blower)
	Relative humidity:	100	% (assumed before blower)
	Air temperature:	120	°F (after blower)
	Relative humidity:	20	% (after blower)
	Design contaminant:	Cis-1,2-DCE	
	Influent concentration:	187.1	µg/L
	Effluent criteria:	< 1	µg/L (assumed)

Other contaminants present:

Acetone	0.1	µg/L
Benzene	0.2	µg/L
Bromomethane	0.1	µg/L
Carbon disulfide	1.2	µg/L
Chloroethane	5.9	µg/L
Chloromethane	0.1	µg/L
1,1-DCA	16.9	µg/L
Ethylbenzene	7.9	µg/L
Isopropylbenzene	42.9	µg/L
Methyl acetate	0.4	µg/L
Methyl cyclohexane	258.1	µg/L
MEK	0.2	µg/L
Methylene chloride	0.1	µg/L
PCE	181.5	µg/L
Toluene	3.6	µg/L
1,1,1-TCA	17.2	µg/L
TCE	5.8	µg/L
Vinyl chloride	9.9	µg/L
Xylenes	25.0	µg/L

Recommendations: Vapor Phase Carbon Adsorbers

Two GPC3's in parallel, each with 200 lbs of carbon

- The following compounds will not be effectively removed by carbon adsorption: acetone, carbon disulfide, chloroethane, chloromethane, methyl acetate, methylene chloride, and vinyl chloride.
- Cis-1,2-DCE will be the next compound to break through and is used as the design contaminant.
- The carbon usage rate is predicted to be 177.207 lbs/day. Both PC3 adsorbers are predicted to last ~ 2 days.

01/05/05

VAPOR-PHASE CARBON MODEL CALCULATIONS

CARBONAIR ENVIRONMENTAL SYSTEMS
2731 NEVADA AVENUE NORTH
NEW HOPE, MN 55427
PHONE: 800-526-4999
FAX: 763-544-2151

```
DESIGN COMPOUD:                              CIS-1,2-DCE
EXPECTED CONC. (UG/L):                           187.100
MODEL CONC. (UG/L):                              260.000
TEMPERATURE (F):                                 120.000 ← AFTER BLOWER
RELATIVE HUMIDITY (%):                            20.000 ← AFTER BLOWER
OPERATING PRESS (MM MERCURY):                    760.000
VAPOR PRESS (MM MERCURY):                        512.186
K VALUE (UMOLE/GM)(L/UMOLE)**1/N:                222.580
1/N VALUE (DIMENSIONLESS):                         0.579
CARBON CAPACITY (%):                               3.953
AIR FLOW RATE (CFM):                             300.000

CARBON USAGE (LBS/DAY):                          177.207
```

Note: The model concentration results from the impact of the other background compounds, which is determined by using a competitive adsorption model.

UG = microgram, UMOLE = micromole

DISCLAIMER: ACTUAL RESULTS MAY VARY SIGNIFICANTLY FROM THE MODEL. THE MODEL IS BASED ON THE ASSUMPTIONS THAT THE FLOW RATE AND INFLUENT CONCENTRATION ARE CONSTANT, AND ONLY THE CONTAMINANTS PROVIDED TO CARBONAIR ARE PRESENT IN THE AIR. VARYING OPERATING CONDITIONS CAN HAVE ADVERSE EFFECTS ON CARBON ADSORPTIVE CAPACITY. THE PREDICTED CARBON USAGE RATE IS NOT GUARANTEED.

Attachment C
DAR Letter Notification

Attachment C

DAR Letter Notification

January 20, 2005

Mr. Sean Snee
New York State Department of Environmental Conservation (NYSDEC)
Division of Air Resources
Region 1 Air Pollution Control Program
Building 40 SUNY
Stony Brook, New York 11790-2356

Subject: Letter of Notification of SVE Treatability Testing
Jackson Steel Superfund Site, Mineola, New York

Dear Mr. Snee:

The purpose of this letter is to notify the Division of Air Resources, Region 1 Air Pollution Control Program (DAR) of a soil vapor extraction (SVE) treatability test that will be conducted at the Jackson Steel Superfund Site in Mineola, New York. This work is being conducted for under a Record of Decision signed by EPA Region II in September, 2004, and is therefore exempt from DAR permit requirements per NYSDEC regulations, Subpart 201-3.3 (29) Exemptions and Trivial Activities. Subpart 201-3.3 (c) states "...The following activities are trivial and are exempt from permitting requirements and do not need to be included in the title V facility permit application:", and Supart 201-3.3 (c) (29) includes "Air strippers and soil vents required under the provisions of an order on consent or stipulation agreement, or in operation at a superfund site."

CH2M HILL is performing the SVE treatability test and submitting this letter on behalf of EPA Region II to provide DAR with sufficient background information, test description, and emission estimates to demonstrate that the treatability test will meet the substantive requirements of applicable emissions regulations, but will not be requesting a formal review.

The Jackson Steel site is an inactive metal forming facility located at 435 First Street in Mineola, North Hempstead, Nassau County, New York (zip code 11501). The entire site is approximately 66,000 square feet or 1.5 acres and consists of one building with a paved parking area. The building, which is currently mostly empty, occupies approximately 42,500 square feet of the property. A paved parking area, approximately 10,000 square feet, is located behind the building . A plan view of the site is presented in Figure 1.

The SVE treatability test will consist of vapor extraction from up to 4 extraction wells located in the parking area. Vacuum influence observations will be conducted at 8 nested vapor monitoring probes and other onsite wells. Extracted soil vapor will be treated using 2 vapor-phase carbon vessels. The treatability test is expected to start operation in March 2005 and will be in operation for up to seven days, operating twelve hours per day. A typical SVE process and instrumentation diagram is presented in Figure 2. The final selection of vendors is in progress and the actual configuration may vary slightly but the emissions will stay the same. Emissions were estimated based on anticipated flow rates and vapor concentrations. Vapor concentrations were estimated based on available soil concentration data using partitioning equations. A data sheet for the SVE treatability test is presented in Attachment A.

Maximum actual annual impact (C_a), maximum potential annual impact (C_p), and maximum short-term impact (C_{st}) concentrations were calculated in accordance with November 12, 1997 DAR-1 Guidelines for the Control of Toxic Ambient Air Contaminants. These values were compared to Short-Term Guideline Concentrations (SGCs) and Annual Guideline Concentrations (AGCs) obtained from the DAR-1 (Air Guide – 1) AGC/SGC Memorandum dated December 22, 2003. The comparison shows that C_a and C_p is less than the AGC and C_{st} is less than the SGC for all contaminants expected to be present in the treated vapor stream during the treatability test. A summary of calculations is presented in Table 1.

The results of the treatability test will be incorporated into the design basis for a larger scale SVE treatability test that will be implemented over the majority of the Jackson Steel Superfund Site during the summer of 2005. Prior to implementation, CH2M HILL will provide a letter notification including emission estimates for the full-scale treatability test to DAR.

If you have any questions or concerns, please do not hesitate to contact me at 802-453-5754.

Sincerely,

CH2M HILL

E. Matt Germon
Project Task Lead

Cc: Christos Tisiamis/EPA Region 2
Juliana Hess/CH2M HILL
Terri Gerrish, P.E./CH2M HILL
CH2M HILL Project Files

TABLE 1

Comparison of Emission Concentrations with Short-term (1-hr) Guideline Concentration/Annual Guideline Concentrations (SGC/AGC)

Jackson Steel Phase 1 SVE Project

Phase 1 Conditions:			
Building height	20 ft	Test duration	7 days
Stack height	24 ft	Operation per day	12 hrs per day
Flow	150 scfm		

	Pre treatment (Uncontrolled)			Post treatment (Controlled)						NYSDEC Guidelines^G	
Chemical	**Extracted Vapor Conc. (ug/m^3)**	**Estimated Total Mass Extracted During Test^A (lb)**	**Estimated Carbon Treatment Efficiency^B**	**Emitted Concentration During Test (ug/m^3)^C**	**Q Hourly Mass Emission Rate (lb/h)**	**Q_a Total mass emitted During Test (lb)**	**C_a (ug/m3)^D**	**C_p (ug/m3)^E**	**C_{ST} (ug/m3)^F**	**SGC (1-hr) (ug/m^3)**	**AGC (annual) (ug/m^3)**
Acetone	20.33	0.001	0%	20.33	0.000	0.001	0.000	0.000	0.031	180,000	28,000
Benzene	29.64	0.001	99%	0.30	0.000	0.000	0.000	0.000	0.000	1,300	0.1
Bromomethane	10.45	0.000	0%	10.45	0.000	0.000	0.000	0.000	0.016	3,900	5
Carbon disulfide	221.35	0.010	0%	221.35	0.000	0.010	0.000	0.005	0.333	6,200	700
Chloroethane	1,117.20	0.053	0%	1,117.20	0.001	0.053	0.000	0.026	1.680	na	10,000
Chloromethane	23.94	0.001	0%	23.94	0.000	0.001	0.000	0.001	0.036	22,000	90
Cyclohexane	4.37	0.000	99%	0.04	0.000	0.000	0.000	0.000	0.000	na	6,000
1,1-Dichloroethane	3,202.07	0.151	99%	32.02	0.000	0.002	0.000	0.001	0.048	na	0.6
Dichloroethylene-1,2 cis	35,554.13	1.678	99%	355.54	0.000	0.017	0.000	0.008	0.535	na	1,900
Ethylbenzene	1,503.47	0.071	99%	15.03	0.000	0.001	0.000	0.000	0.023	54,000	1,000
Hexanone-2 (methyl n-butyl ketone)	3.99	0.000	99%	0.04	0.000	0.000	0.000	0.000	0.000	4,000	48
Isopropylbenzene	8,143.02	0.384	99%	81.43	0.000	0.004	0.000	0.002	0.122	na	400
Methyl acetate	66.69	0.003	0%	66.69	0.000	0.003	0.000	0.002	0.100	76,000	1,400
Methyl cyclohexane	49,032.35	2.314	99%	490.32	0.000	0.023	0.000	0.011	0.737	na	3,800
Methyl ethyl ketone (2-butanone)	31.92	0.002	99%	0.32	0.000	0.000	0.000	0.000	0.000	59,000	5,000
Methyl isobutyl ketone (4-methyl-2-pentanone)	0.00	0.000	99%	0.00	0.000	0.000	0.000	0.000	0.000	31,000	3,000
Methyl tertiary butyl ether (MTBE)	0.57	0.000	99%	0.01	0.000	0.000	0.000	0.000	0.000	na	3,000
Methylene chloride	27.74	0.001	0%	27.74	0.000	0.001	0.000	0.001	0.042	22,000	90
Tetrachloroethylene	34,483.29	1.627	99%	344.83	0.000	0.016	0.000	0.008	0.519	1,000	1.0
Toluene	681.72	0.032	99%	6.82	0.000	0.000	0.000	0.000	0.010	37,000	400
1,1,1-Trichloroethane	3,267.43	0.154	99%	32.67	0.000	0.002	0.000	0.001	0.049	na	1.4
Trichloroethylene	1,096.11	0.052	99%	10.96	0.000	0.001	0.000	0.000	0.016	54,000	0.5
Trichlorofluoromethane	0.00	0.000	99%	0.00	0.000	0.000	0.000	0.000	0.000	560,000	na
vinyl chloride (chloroethene)	1,873.78	0.088	0%	1,873.78	0.001	0.088	0.000	0.043	2.818	180,000	0.1
Xylenes, total	4,740.88	0.224	99%	47.41	0.000	0.002	0.000	0.001	0.071	4,300	100

Notes:

A - Pilot test will be operated at 150 scfm for up to 7 days, 12 hours per day.

B - A destruction efficiency of 99% is assumed for all compounds that can be removed by carbon treatment.

C - Emitted Concentration = Extracted Vapor Concentration x (1 - Treatment Efficiency)

D - Calculated according to New York State Department of Conservation, DAR-1, Guidelines for the Control of Toxic Ambient Air Contaminants, Appendix B, Section III.A.2.

C_a (ug/m^3) = (6.0 x Q_a)/(he$^{2.25}$)

Values were compared to AGC limits and there were no exceedances.

E - Calculated according to New York State Department of Conservation, DAR-1, Guidelines for the Control of Toxic Ambient Air Contaminants, Appendix B, Section III.A.3.

C_p (ug/m^3) = (52500 x Q)/(he$^{2.25}$)

Values were compared to AGC limits and there were no exceedances.

F - Calculated according to New York State Department of Conservation, DAR-1, Guidelines for the Control of Toxic Ambient Air Contaminants, Appendix B, Section III.A.5.

C_{ST} (ug/m^3) = C_p x 65

Values were compared to SGC limits and there were no exceedances.

G - SGC/AGC values are adopted from New York State Department of Environmental Conservation, DAR-1 AGC/Tables, Division of Air Resources, Air Toxics Section, December 22, 2003.

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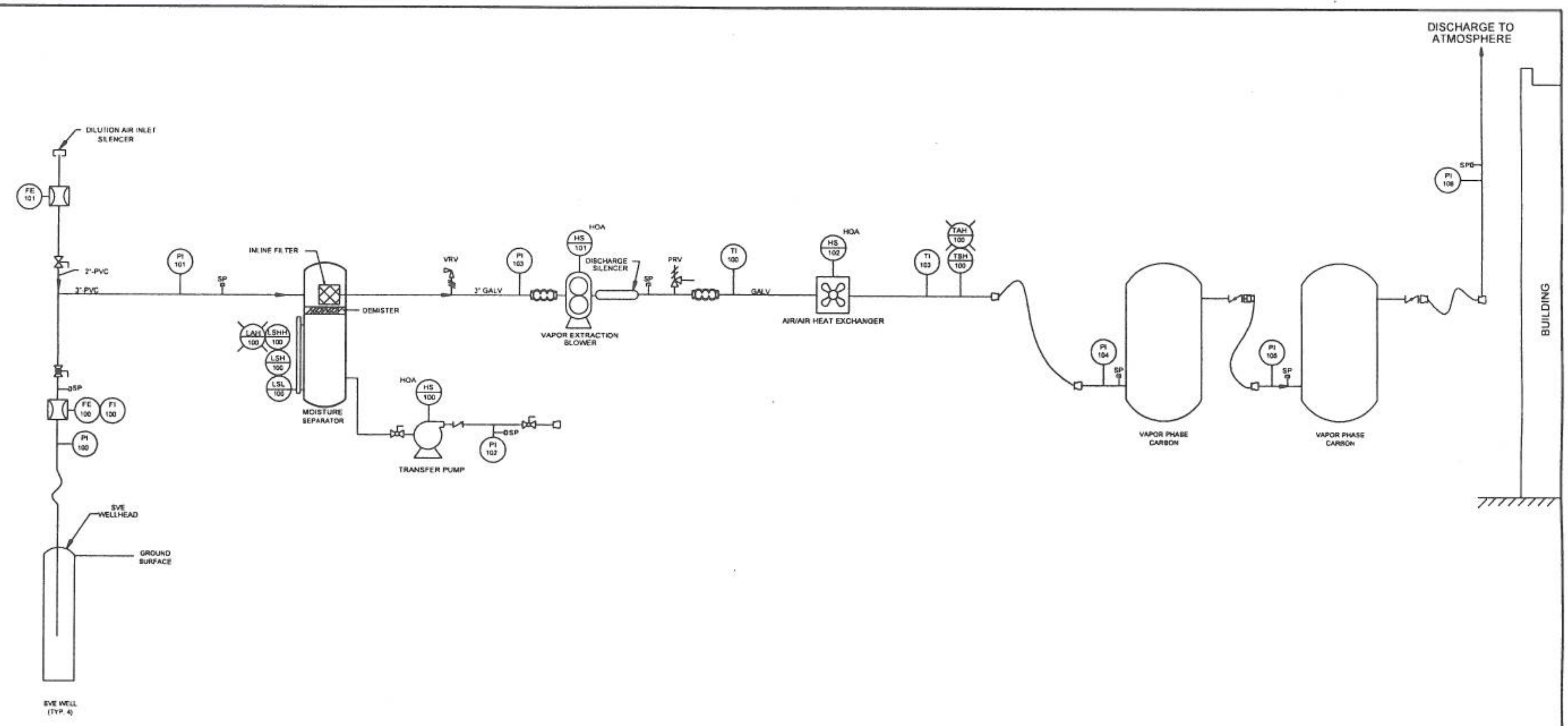

LEGEND

PI	PRESSURE INDICATOR
FE	FLOW ELEMENT
FI	FLOW INDICATOR
DPI	DIFFERENTIAL PRESSURE INDICATOR
⧖	GATE VALVE/BUTTERFLY VALVE
⧗	BALL VALVE
HS	HAND SWITCH
LAH	LEVEL ALARM HIGH
LSHH	LEVEL SWITCH HIGH-HIGH
LSH	LEVEL SWITCH HIGH
LSL	LEVEL SWITCH LOW
TI	TEMPERATURE INDICATOR
TAH	TEMPERATURE ALARM HIGH
TSH	TEMPERATURE SWITCH HIGH
SP	SAMPLING PORT

DISCHARGE TO ATMOSPHERE

Figure 2

SVE PROCESS AND INSTRUMENTATION DIAGRAM

Jackson Steel Superfund Site
Mineola, NY

CH2MHILL

ATTACHMENT A

SVES Data Sheet

TO: **DAR, Region 1 _____**

FROM: **CH2M HILL on behalf of EPA Region II**

1. Reason for Submittal:
   X Notice of Operation
   ☐ Notice of Removal of Emission Control Equipment

2. Spill Name: Jackson Steel Superfund Site
   Spill Location: 435 1st Street, Mineola, NY

3. Spiller: EPA Region II
   Address:
   Christos Tsiamis, Project Manager
   EPA Region II, NY Branch
   290 Broadway 20th Floor
   New York, NY 10007

4. Spill Number: NA PIN Number: NA

5. Start-Up Date: March 2005
   Mo/day/yr

6. Estimated Project Duration: 7 ~~Months or Years~~ Days

7. Emission Point:
   a. Emission I.D. Number: 1
   b. Ground Elevation Above Sea Level: ~147 FT
   c. Stack Height: 24 FT
   d. Height Above Nearest Structure: 4 FT
   e. Stack Inside Dimensions: 3-inch PVC SCH 40 ~~FT~~
   f. Air Exit Temperature: 120 F
   g. Air Flow-rate: 150 CFM
   h. Air Exit Velocity: 50.9 FT/SEC
      $= \frac{\text{Air Flow-rate in CFM}}{\text{Cross-sectional Area of Stack in FT}^2 \times 60}$
   i. Benzene Concentration in Air Influent: 29.64 UG/M^3
      _____ LB/HR
      _____ PPM-V
   j. Distance From Base of Stack to Nearest On-Site Bldg: 5 FT
   k. Distance From Base of Stack to Nearest Off-Site Bldg: 75 FT

8. Operation Time 7 days
   a. Hours/Day: 12

b. Days/Year: 7
c. % Operation by Season: 0% Winter 0% Summer
100% Spring 0% Fall

9. Process Description: PD Blower, Heat exchanger, 2 vapor-phase granular activated carbon vessels in series. See Figure 2.

10. Emission Controls
☐ Not Needed Based on Analysis of Pilot Test Data
☐ Not Needed Based on Analysis of Operating Data
X Described Below

11. Control Equipment
a. I.D. Number: NA (to be identified on site plan as well)
b. Control Type:
☐none ☐thermal afterburner
X activated bed adsorber ☐catalytic unit
☐other, explain: _____
c. Manufacturer's Name: Not yet known
d. Model Number: Not yet known
e. Disposal of Collected Contaminants:
☐landfill off-site ☐recycled on-site
☐recycled in the process ☐public sewer
X other, explain: Regenerated at approved facility upon completion of pilot test
f. Date Emission Control Operations Began: March/2005 _____
g. Expected Useful Life: 7 ~~Months or Years~~ Days

12. Contaminant: (**X** Multiple contaminants – see attached) See Table 1 for complete list
a. Name: _____
a. CAS Number: _____
b. Control Equipment Input (= 7i.): _____ LBS/HR (or UG/m^3 or PPM-V)
c. Control Equipment Efficiency: _____%
d. Control Equipment Output (= (1-12d.) x 12c.): _____ LBS/HR (or UG/m^3 or PPM-V)
e. Permissible Air Output Conc.: _____ LBS/HR (or UG/m^3 or PPM-V)

13. Fuels for Combustion Vented to the Same Emission Point
a. Fuel
X None ☐Oil ☐Gas
☐Other Explain: _____
b. Fuel Type
☐#2 Fuel ☐Natural Gas
☐#4 Fuel ☐LP Gas
☐Diesel Fuel ☐Other, Explain: _____
c. Amount:_____ x 10^3 GALS/YR (oil)
_____ x 10^3 FT^3/YR (gas)
c. For Oil Only, Sulfur Content: _____% By Weight
d. For Gas Only, Heating Value: _____BTU/FT^3

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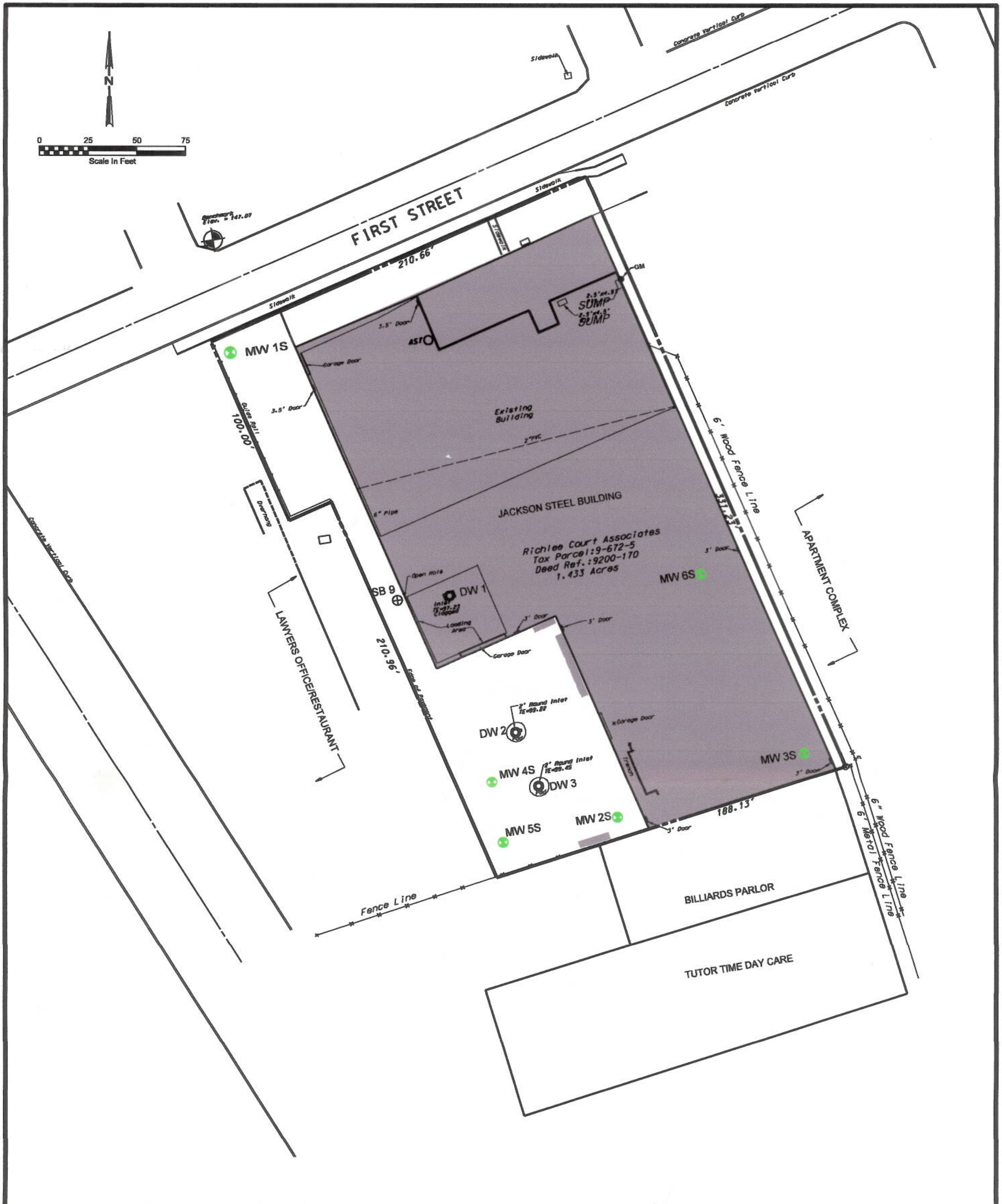

LEGEND

- 🟢 SHALLOW MONITORING WELL
- DW ○ DRYWELL
- ☐ SUMP
- ▨ DRUM STORAGE AREA (LOCATIONS ARE APPROXIMATE)
- ○ ABOVE-GROUND STORAGE TANK
- GM ⊗ GAS METER
- ▪▪ ―― PROPERTY BOUNDARY

GENERAL NOTES

1. BEARINGS ARE FROM A BOUNDARY SURVEY DONE PRIOR TO ROTATION OF THE DRAWINGS TO STATE PLANE COORDINATES.
2. NORTH BASED ON STATE PLANE COORDINATES
NEW YORK LONG ISLAND ZONE - NAD 83
DETERMINED BY CODE BASED GPS
3. BENCHMARK - SURVEY DISK (G 6 USE) SET IN
THE TOP OF A CONCRETE MONUMENT ALONG NORTHER BLVD.
OPPOSITE SHELTER ROCK RD
NGVD 29
ELEV. = 131.33
4. ⊕ SITE BENCHMARK - MAG NAIL T-1
ELEV. = 147.07

SOURCE: DRAWING PROVIDED BY RETTEW ASSOCIATES, INC.; JUNE 5, 2002

Figure 1
SITE PLAN
Jackson Steel RI/FS
Mineola, NY

CH2MHILL

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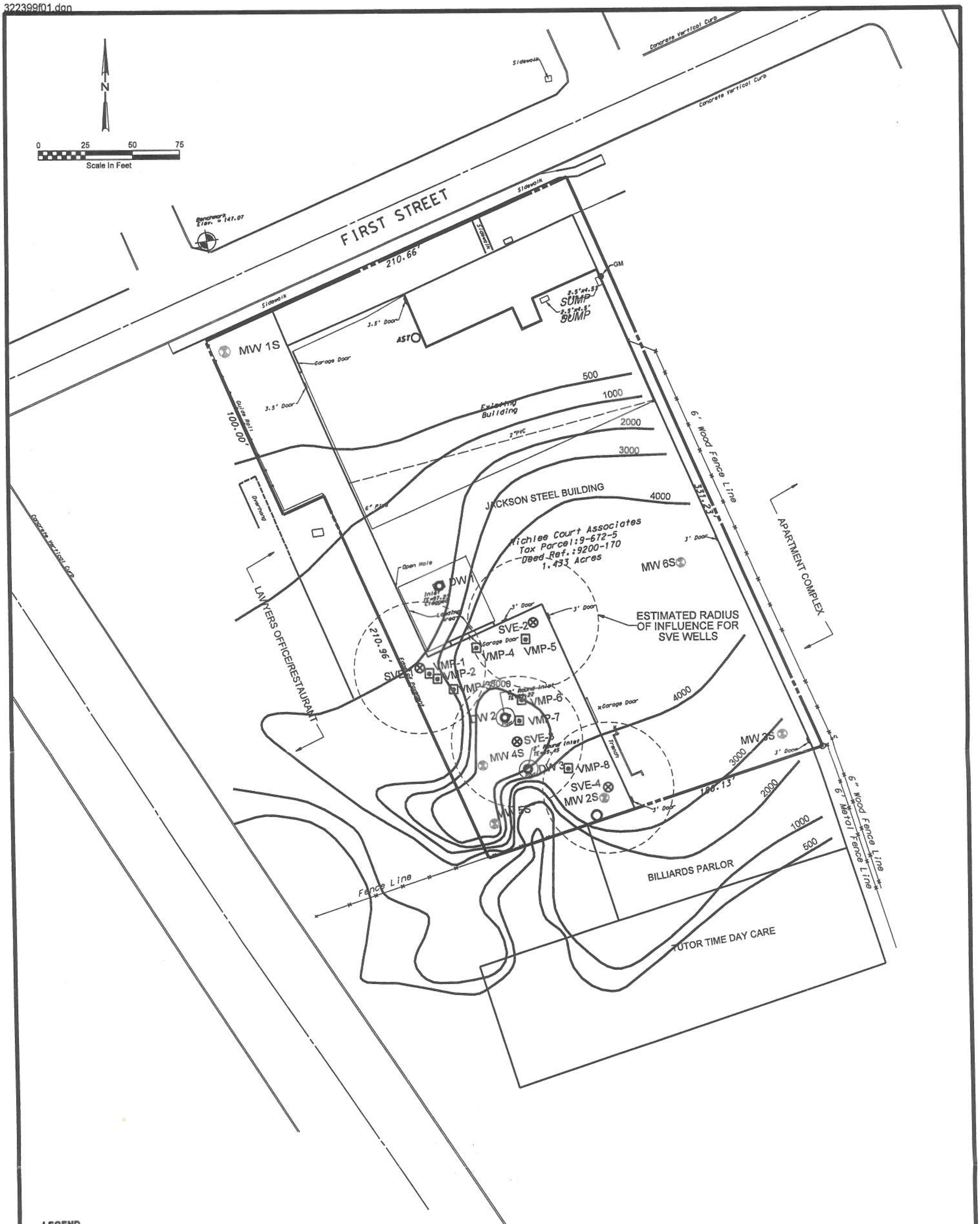


LEGEND

- MONITORING WELL
- ⊗ SVE WELL
- ▣ VAPOR MONITORING PROBE (NESTED)
- ○ BUILDING SLAB VENT

1000 - PCE SOIL GAS ISO CONCENTRATION CONTOUR (ppbv), APRIL 2002

GENERAL NOTES

LAYOUT ASSUMES MW-2S, MW-4S AND MW-5S ARE PARTIALLY SCREENED IN VADOSE ZONE.

ALL SOIL GAS SAMPLES USED TO DEVELOP ISO-CONCENTRATION CONTOURS WERE COLLECTED AT 5-FEET BELOW GROUND SURFACE IN APRIL 2002 BY EPA.

SOURCE: DRAWING PROVIDED BY RETTEW ASSOCIATES, INC.; JUNE 5, 2002

Figure 2
PHASE 1 SVE PILOT TEST LAYOUT
Jackson Steel Superfund Site
Mineola, NY

CH2MHILL

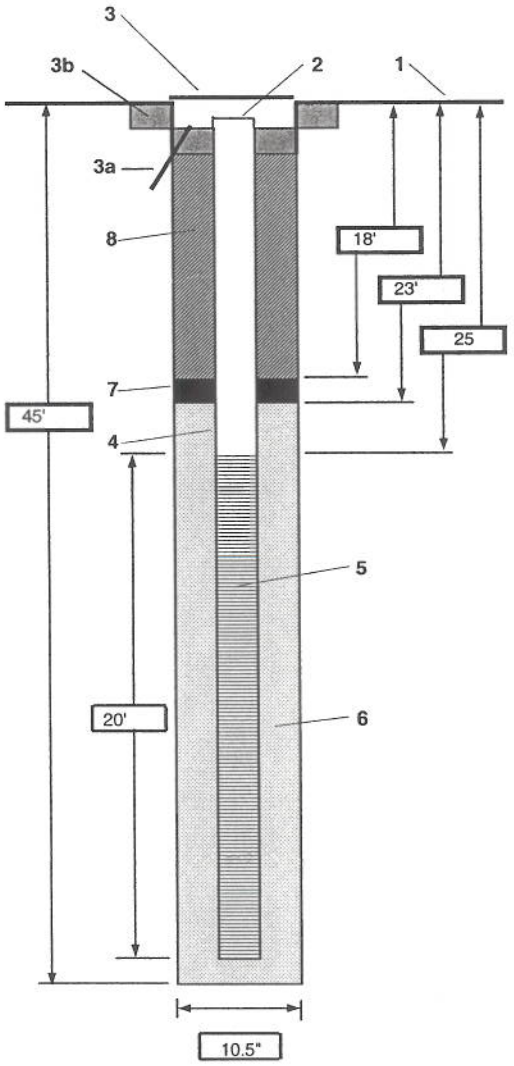
CH2MHILL

SHEET 1 OF 1

PROJECT : Jackson Steel Pre-Remedial Design

LOCATION : Mineola, NY

DRILLING METHOD AND EQUIPMENT USED :



1- Ground elevation at well	TBD BY Surveyor
2- Top of casing elevation	TBD BY Surveyor
3- Wellhead protection cover type	Flush Mount
a) drain tube?	No
b) pad dimensions	No Pad, loosely set flushmount cover in asphalt
4- Dia./type of well casing	4" Schedule 40 PVC
5- Type/slot size of screen	4"Schedule 40 PVC, 60 Slot
6- Gravel Pack	Global #4 or Equivalent
7- Bentonite Seal	Granular
8- Well Gravel	Same as Gravel Pack

FIGURE 3

SVE Well Construction Diagram (proposed)

Phase 1 SVE Treatability Test Design Basis

Jackson Steel Superfund Site, Mineola, NY

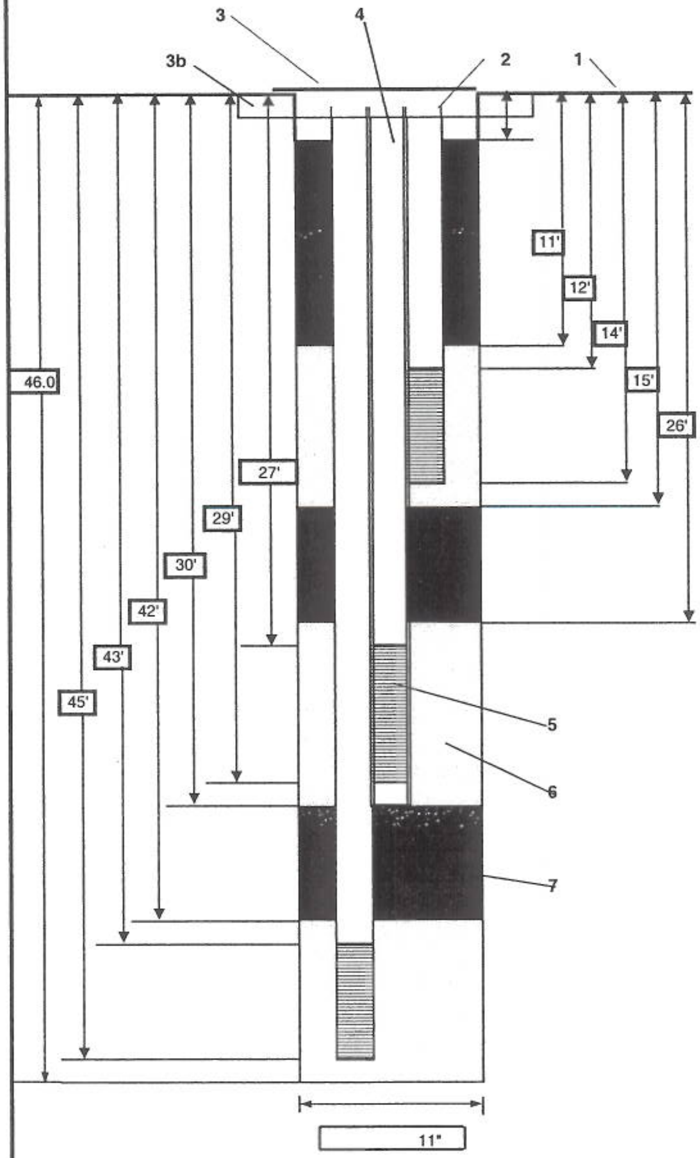
CH2MHILL

SHEET 1 OF 1

PROJECT : Jackson Steel Pre-Remedial Design

LOCATION : Mineola, NY

DRILLING METHOD AND EQUIPMENT USED : 6-1/4" Hollow Stem Auger



1- Ground elevation at well	TBD BY Surveyor
2- Top of casing elevation	TBD BY Surveyor (At least 6" Below Cover)
3- Wellhead protection cover type	Flush Mount
a) concrete pad dimensions	3' x 3' Square
4- Dia./type of well casing	1" Schedule 40 PVC
5- Type/slot size of screen	1"Schedule 40 PVC, 60 Slot
6- Gravel Pack	Global #4 or Equivalent
7- Bentonite Seal	Granular

FIGURE 4

VMP Well Construction Diagram (proposed)

Phase 1 SVE Treatability Test Design Basis

Jackson Steel Superfund Site, Mineola, NY

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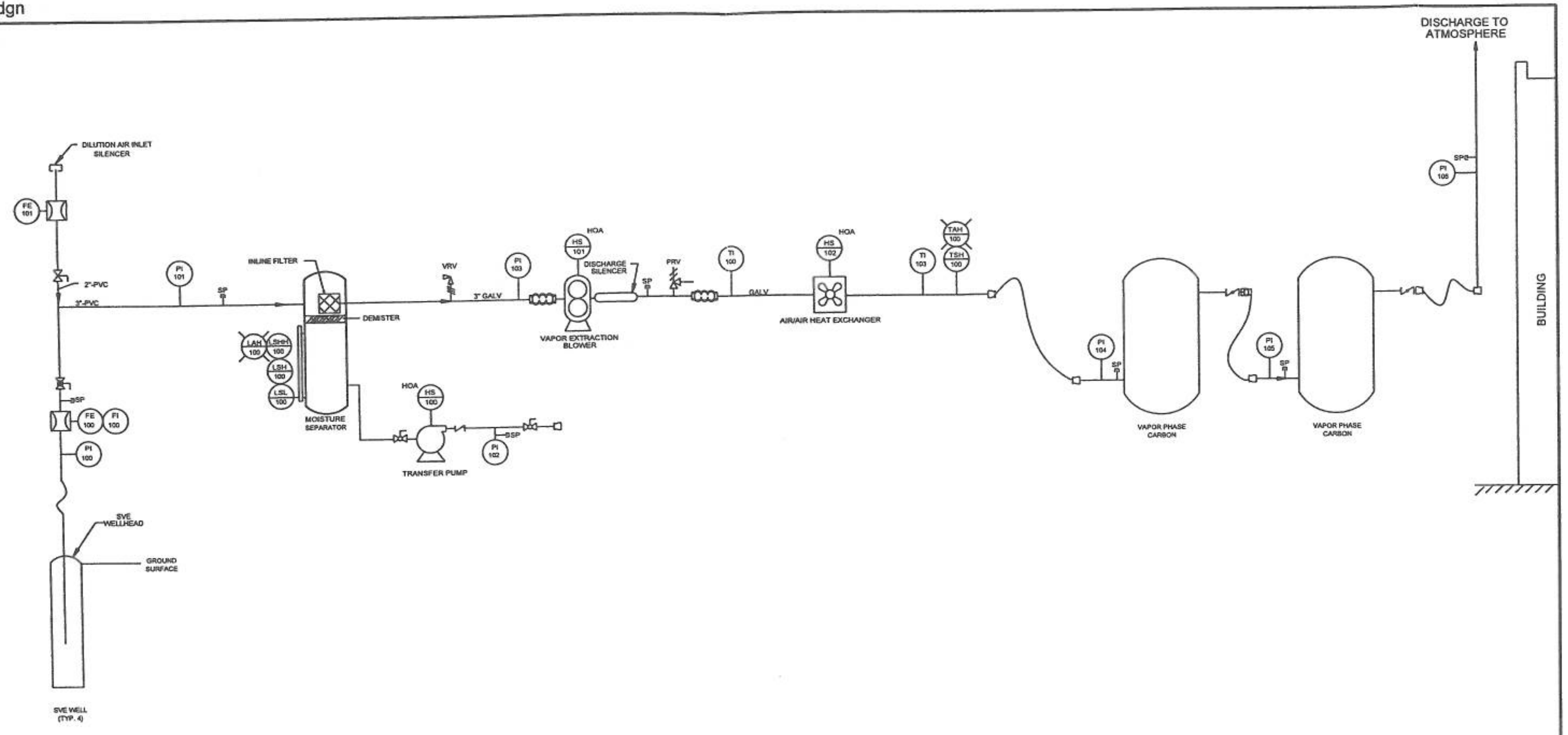

LEGEND

- PI PRESSURE INDICATOR
- FE FLOW ELEMENT
- FI FLOW INDICATOR
- DPI DIFFERENTIAL PRESSURE INDICATOR
- GATE VALVE/BUTTERFLY VALVE
- BALL VALVE
- HS HAND SWITCH
- LAH LEVEL ALARM HIGH
- LSHH LEVEL SWITCH HIGH-HIGH
- LSH LEVEL SWITCH HIGH
- LSL LEVEL SWITCH LOW
- TI TEMPERATURE INDICATOR
- TAH TEMPERATURE ALARM HIGH
- TSH TEMPERATURE SWITCH HIGH
- SP SAMPLING PORT

DISCHARGE TO ATMOSPHERE

Figure 5

TYPICAL SVE PROCESS AND INSTRUMENTATION DIAGRAM

Jackson Steel Superfund Site
Nineola, NY

CH2MHILL

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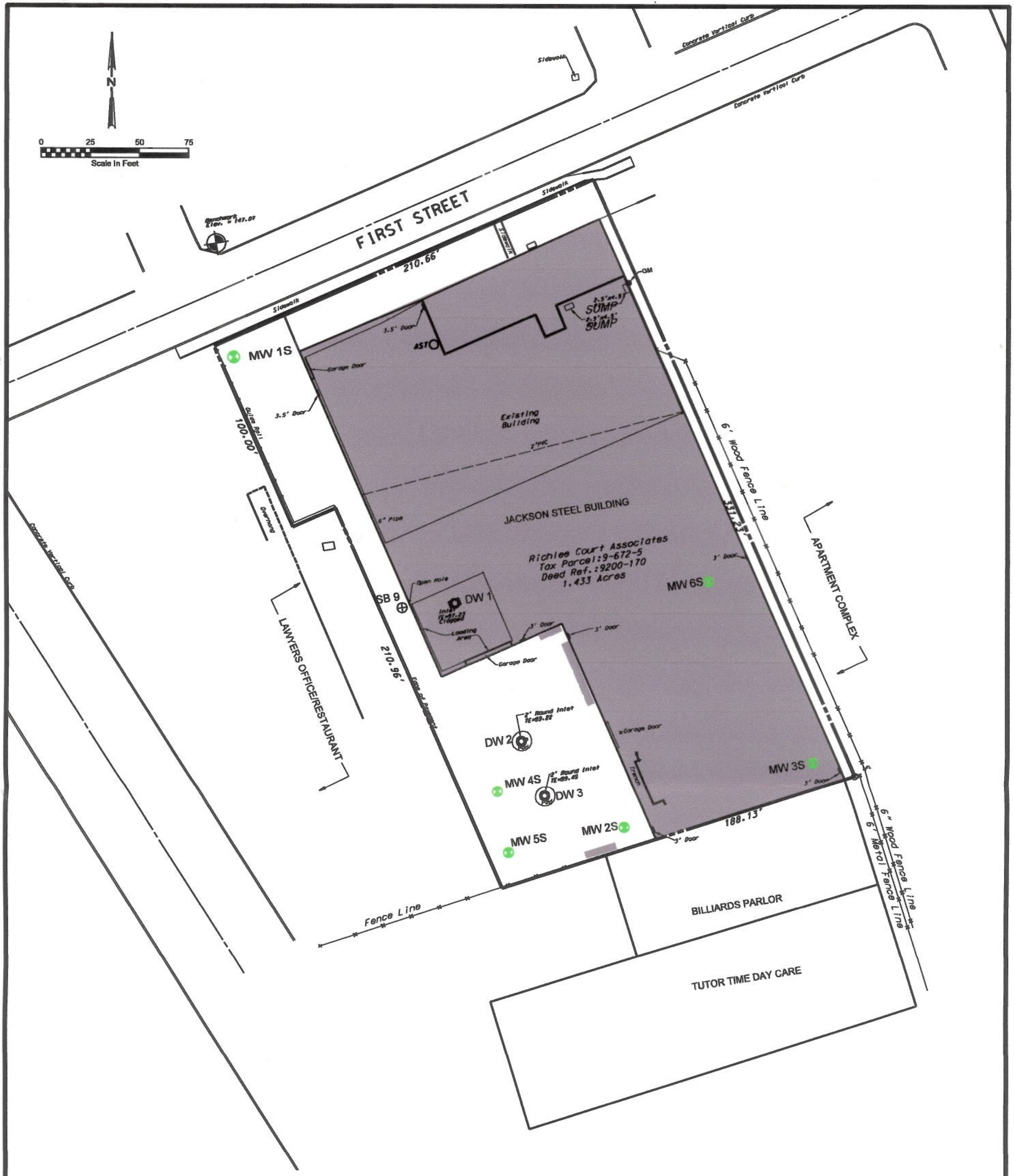

LEGEND

- ● SHALLOW MONITORING WELL
- DW ○ DRYWELL
- ☐ SUMP
- ▨ DRUM STORAGE AREA (LOCATIONS ARE APPROXIMATE)
- ○ ABOVE-GROUND STORAGE TANK
- GM ⊗ GAS METER
- ▬ PROPERTY BOUNDARY

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SOURCE: DRAWING PROVIDED BY RETTEW ASSOCIATES, INC.; JUNE 5, 2002

Figure 1
SITE PLAN
Jackson Steel RI/FS
Mineola, NY

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