

12/1/92-00591

**FINAL**  
**AIR SAMPLING PROGRAM**  
**PROJECT PLAN ADDENDUM**  
**REMEDIAL INVESTIGATION/  
FEASIBILITY STUDY**  
**CAMP ALLEN LANDFILL**  
**NAVAL BASE, NORFOLK, VIRGINIA**  
**CONTRACT TASK ORDER 0084**

*Prepared for:*

**DEPARTMENT OF THE NAVY**  
**ATLANTIC DIVISION**  
**NAVAL FACILITIES**  
**ENGINEERING COMMAND**  
*Norfolk, Virginia*

*Under the:*

**LANTDIV CLEAN Program**  
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## TABLE OF CONTENTS

	<u>Page</u>
<b>1.0 INTRODUCTION</b> .....	<b>1-1</b>
1.1 Previous Investigation .....	1-1
1.2 Description and Purpose of the Current Study .....	1-4
<b>2.0 BACKGROUND AND SETTING</b> .....	<b>2-1</b>
<b>3.0 INVESTIGATIVE TASKS</b> .....	<b>3-1</b>
3.1 General Overview .....	3-1
3.2 Air Sampling Locations: Brig Facility (Area A) .....	3-2
3.3 Air Sampling Locations: Camp Allen Elementary School (Area B) ...	3-2
3.4 Ambient Air Sampling Locations .....	3-8
3.5 Analytical Methodology .....	3-10
<b>4.0 SAMPLING AND ANALYSES PLAN</b> .....	<b>4-1</b>
4.1 Purpose and Method .....	4-1
4.2 Sample Numbering .....	4-1
4.3 Quality Assurance/Quality Control .....	4-2
<b>5.0 QUALITY ASSURANCE PROJECT PLAN</b> .....	<b>5-1</b>
5.1 Introduction and Scope .....	5-1
5.2 Quality Assurance Objectives for Data Measurement .....	5-1
5.3 Sampling Procedure .....	5-3
5.4 Analytical Procedures .....	5-3
5.5 Quality Assurance and GC/MS/Scan System Performance Criteria ...	5-5
5.6 Calibration Procedures and Frequency .....	5-5
5.7 Internal Quality Control Checks .....	5-7
<b>6.0 HEALTH AND SAFETY PLAN</b> .....	<b>6-1</b>
6.1 Site Entry Objectives .....	6-1
6.2 Site Organization and Coordination .....	6-1
6.3 Site Control .....	6-2
6.4 Hazard Evaluation .....	6-2
6.5 Emergency Services Location .....	6-2
<b>REFERENCES</b> .....	<b>R-1</b>

## LIST OF TABLES

<u>Number</u>		<u>Page</u>
3-1	Air Sampling Locations: Brig Facility (Area A) .....	3-3
3-2	Air Sampling Locations: Camp Allen Elementary School (Area B) .....	3-9
3-3	Ambient Air Sampling Locations: Camp Allen Landfill (Areas A and B) ...	3-12
5-1	Target Analytes and Detection Limits .....	5-4
5-2	4-Bromofluorobenzene Key Ions and Ion Abundance Criteria .....	5-6
5-3	Frequencies of Internal Laboratory Quality Control Checks .....	5-8

## LIST OF FIGURES

<u>Number</u>		<u>Page</u>
1-1	Base Location Map .....	1-2
1-2	Site Location Map .....	1-3
3-1	Air Sampling Locations (First Floor) and Utility Detail , Camp Allen Brig Facility Area A .....	3-4
3-2	Air Sampling Locations (Second Floor), Camp Allen Brig Facility Area A ..	3-5
3-3	Air Sampling Locations (Third Floor), Camp Allen Brig Facility Area A ...	3-6
3-4	Air Sampling Locations/Utility Detail, Camp Allen Elementary School (Area B) .....	3-7
3-5	Ambient Air Sampling Locations, Camp Allen Landfill Areas A and B .....	3-11
6-1	Emergency Services Map .....	6-3

## LIST OF APPENDICES

- A Use of Annual Wind Rose for Climatic Applications
- B Compendium Method TO-14
- C Canister Sampling Data Sheets

## 1.0 INTRODUCTION

This document presents the Final Air Sampling Program Project Plan Addendum for Contract Task Order (CTO) 0084, Remedial Investigation/Feasibility Study (RI/FS) for the Camp Allen Landfill Site (Areas A and B), Naval Base, Norfolk, Virginia. This plan is being introduced as an addendum to the Project Plans originally prepared under this CTO for the Camp Allen RI/FS. Figure 1-1 presents a Base Location Map.

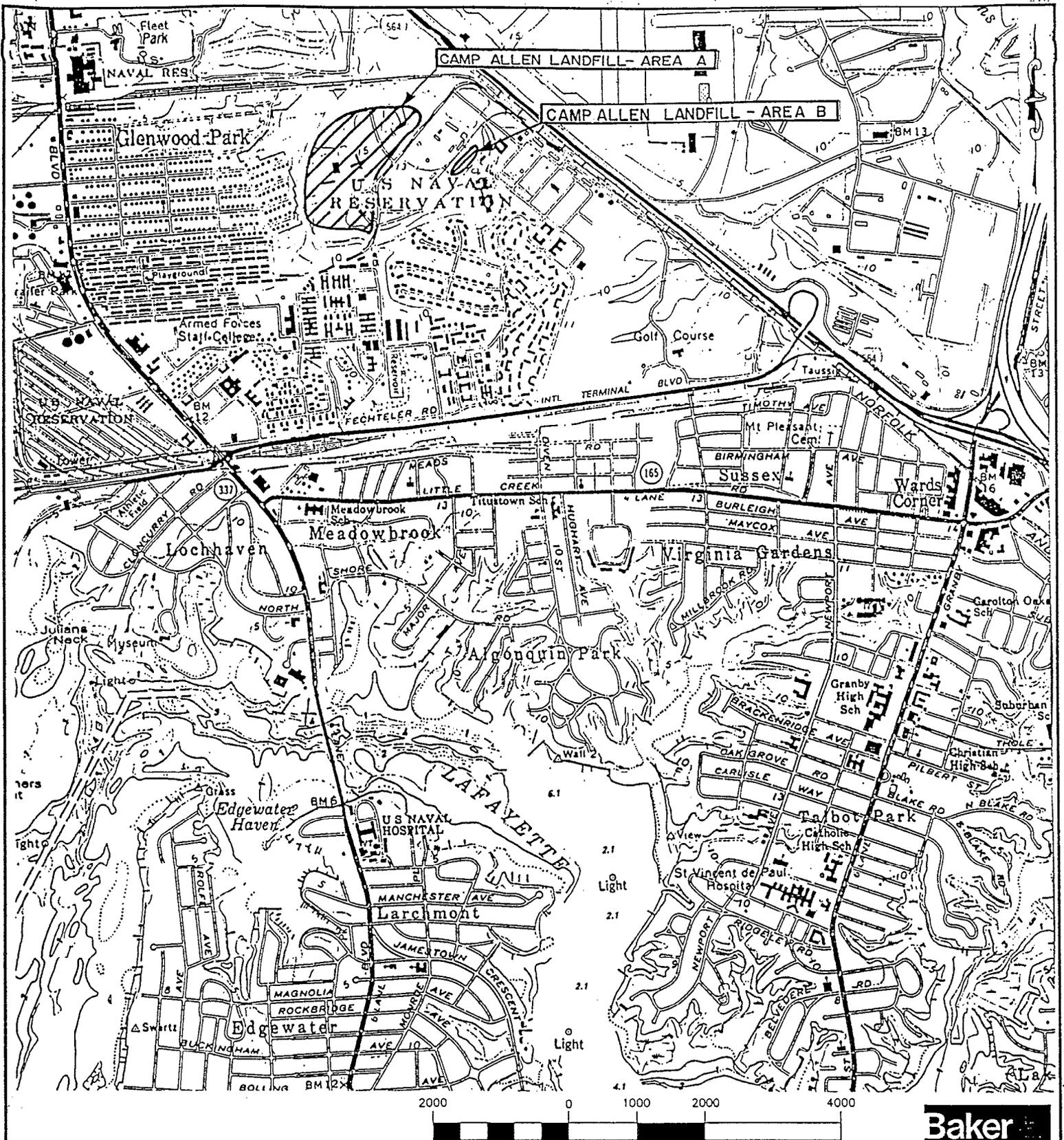
The primary study area will focus on the Brig Facility (Area A) due to the proximity of potential receptors. Potential concerns with regard to the Brig have been identified based on evaluation of the data collected during the review of historical information and the preliminary findings from the Remedial Investigation. In addition, the Camp Allen Elementary School will also be investigated as a precautionary measure. The elementary school has been selected for air sampling due to its proximity to the landfill (Area B) and the nature of the potential receptors. Figure 1-2 presents a Site Location Map.

### 1.1 Previous Investigation

In 1983, a Site Suitability Assessment was performed by Malcolm Pirnie, Inc., for a proposed Brig expansion. Groundwater monitoring wells (11), soil borings (20) and gas monitoring stations (9) were installed in the vicinity of the Brig. The gas monitoring stations were installed to measure potential methane releases during construction operations.

From November 29 through December 1, 1983, gas monitoring was performed for thirty volatile organic priority pollutants to determine the existence, production and release of gases at the Camp Allen Landfill. One sample from each of the gas monitoring stations was collected using a charcoal tube and low flow pump, and transported to Gollob Analytical Service laboratory for analysis. No pollutants were found to be above the established analytical detection limits.

In addition, field screening tests were performed to determine the levels of volatile organic gases, hydrogen sulfide, methane, combustible gas, and oxygen deficiencies. Methane was the only gas detected in measurable concentrations, and was generally assumed to be due to natural organic degradation. Concentrations of methane gas detected at three of the monitoring stations were considered trace amounts (less than 300 parts per million); however, one station (B-8) exhibited a substantial quantity of methane (> 1000 ppm). The significant

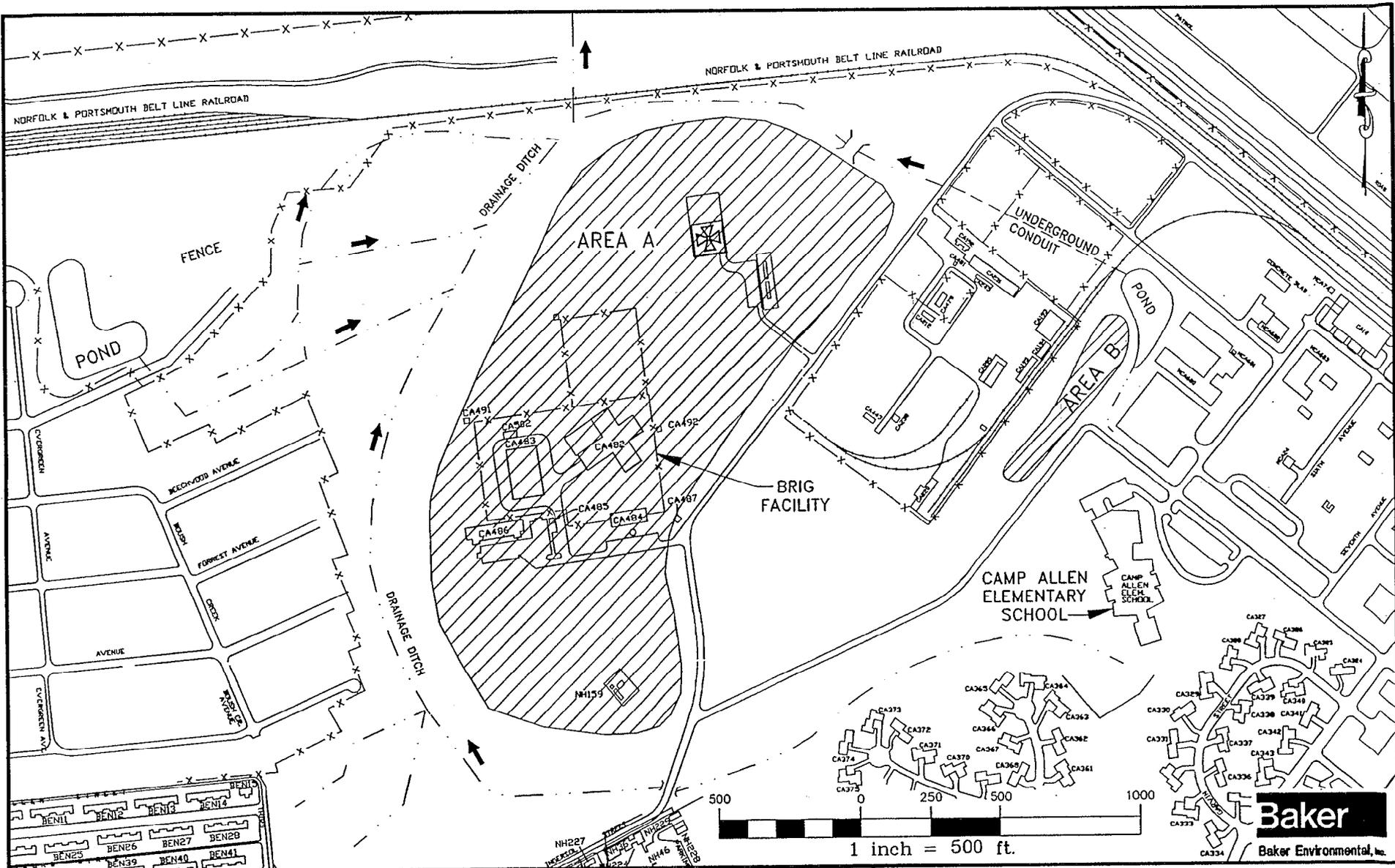


SOURCE: U.S.G.S. TOPOGRAPHIC MAP

1 inch = 2000 ft.

**Baker**  
Baker Environmental, Inc.

FIGURE 1-1  
 BASE LOCATION MAP  
 CAMP ALLEN LANDFILL  
 AREAS A and B  
 NORFOLK NAVAL BASE  
 NORFOLK, VIRGINIA



**LEGEND**

 ASSUMED LANDFILL BOUNDARY  
 SURFACE WATER FLOW DIRECTION

SOURCE: LANTDIV, OCTOBER 1991

**FIGURE 1-2**  
**SITE LOCATION MAP**  
**CAMP ALLEN LANDFILL AREAS A & B**  
 NORFOLK NAVAL BASE  
 NORFOLK, VIRGINIA

amount of methane at station B-8 could not conclusively be attributed to the landfill material due to potential leaks in a sewer pipe and/or grease trap adjacent to the monitoring location. Also, during field screening activities, a positive reading for toxic gas was indicated at station B-18. Malcolm Pirnie recommended a further investigation at stations B-8 and B-18 in order to identify the source, but no additional air sampling has been performed to date.

In April of 1991, a report was published by CH<sub>2</sub>M Hill, identifying the results of a soil gas survey performed within the Area B Landfill. The results of this survey indicated the presence of cycloalkanes/alkenes, chlorinated hydrocarbons, and aromatic hydrocarbons in the soil at Area B. Geoprobe investigations performed in April of 1992 by Baker Environmental, Inc., in the vicinity of the elementary school, also indicated the presence of volatile organics in the shallow groundwater.

## **1.2 Description and Purpose of the Current Study**

The generation of emissions from the Camp Allen Landfill Site (Areas A and B) depends on several key chemical and physical properties of the waste materials disposed at the site. These data can be used to help estimate the potential air emissions from the site, given the critical factors imposed by the site.

For volatile compounds, the rate limiting factor is the movement of vapors through the soil. Volatilization into the soil pore spaces and transfer from the soil-gas into the atmosphere once the soil/air interface has been reached, is usually quite rapid. The generation of leachate from landfills can accelerate the pollutant migration into the groundwater below and provide an additional source of air emissions resulting from the volatilization of dissolved contaminants in the groundwater. The contaminated groundwater can also transfer contaminants into the soil-gas and hence the atmosphere.

The purpose of the current study at the Camp Allen Landfill Site (Areas A and B) is to provide analytical support in the assessment of potential health risks from certain volatile organic compounds (as identified in previous studies) which may become airborne, and escape from the confines of the landfill. The results of the air monitoring data will be evaluated against federal and state Applicable or Relevant and Appropriate Requirements (ARARs), to determine if there is a threat to personnel within the Brig Facility and the elementary school.

In addition to samples obtained within the Brig and elementary school, samples of the ambient air will be collected in areas located upwind and downwind of these facilities, as well as, a point source air sample located adjacent to the Brig and downwind of a previously installed gas monitoring station.

## 2.0 BACKGROUND AND SETTING

The background and setting of the Camp Allen Landfill, Brig Facility and elementary school area are detailed in the Final Project Plans (April 23, 1992). This section adds references to air-related issues only. At present, the majority of Areas A and B are grass covered to minimize the potential for surface erosion. Area A incorporates the Navy Brig Facility in addition to a heliport built over a portion of the landfill. Area B consists of a field located between the Base Salvage Yard operation and the elementary school.

### 3.0 INVESTIGATIVE TASKS

#### 3.1 General Overview

Emission of air pollutants from the Camp Allen Landfill is dependent on the chemical and physical properties of the disposed wastes and on the landfill design components which may have been implemented to reduce air emissions. The relative position of the landfill waste will influence the potential for air emissions and, thus, the air pathway analyses objectives. The amount of waste present at specific locations and the nature of the waste will strongly influence the baseline emissions at the site. The methane gases generated from the landfill can often increase the migration potential of high concentrations of non-methane hydrocarbons by acting as a carrier medium during bulk flow transport of these contaminants.

Site characterization activities performed during the initial field investigation were intended to determine potential or existing contaminant migration by direct contact, surface water and groundwater pathways. Each pathway, including air, represents a potential route of exposure to the public and the general environment.

Where gas monitoring stations, (e.g., gas venting systems) have been installed, volatile emissions from the controls are likely to be higher than emissions from the site surface. The lateral migration of solid and liquid wastes into the surrounding soils and beneath the containment area can create large areas of contaminated subsurface soils. The contaminated soils then represent a source of potential air emissions via the transfer of contaminants into the atmosphere at the surface soil/atmosphere interface.

Air pathways will be difficult to ascertain as it is anticipated that the site will emit little perceptible air emissions in its baseline or undisturbed state. Exposure to the elements over time often causes surface materials to develop a type of surface crust which tends to also impede volatile emissions. In addition, the location of sampling points must account for less than optimum climate conditions. Therefore, all sample locations have been estimated based on theoretical air pathway analyses (without the application of modeling), scientific data and historical information. The baseline emission locations have been established based on variations attributed to the type of disposed waste, the operating practices, the age of the facilities, facility designs, and utility locations. Selected/recommended air sampling points, and rationale for their selection, are presented in the following sections. Given site conditions

and air sampling requirements, program results will include discussions of potential limiting factors.

### **3.2 Air Sampling Locations: Brig Facility (Area A)**

Air sampling locations within the buildings associated with the Brig Facility and the surrounding area have been selected based on the following information:

- The amount of personnel activity taking place within each building, the estimated duration and acute versus chronic exposure conditions.
- The proximity of the buildings to various "hot spots" determined from soil/groundwater analysis and gas monitoring stations, as volatile emissions from control points are likely to be higher than emissions from the site surface.
- The flow of groundwater and potential migration of contaminants both horizontally and vertically, as contaminated soils represent a source of potential air emissions via the transfer of contaminants in the atmosphere.
- The network of various utilities (i.e., sanitary sewers, storm sewers, steam lines, electric lines, and water lines) due to potential air pathway emissions resulting from the utility design. In addition to utility intakes, the construction of the building foundations and wall sections were preliminarily evaluated for potential air pathways for volatile emissions.

In order to adequately characterize the buildings associated with the Brig Facility the sampling scheme itemized in Table 3-1 has been designed. Figures 3-1, 3-2, and 3-3 present proposed SUMMA canister locations for the Brig Facility first floor, second floor, and third floor, respectively.

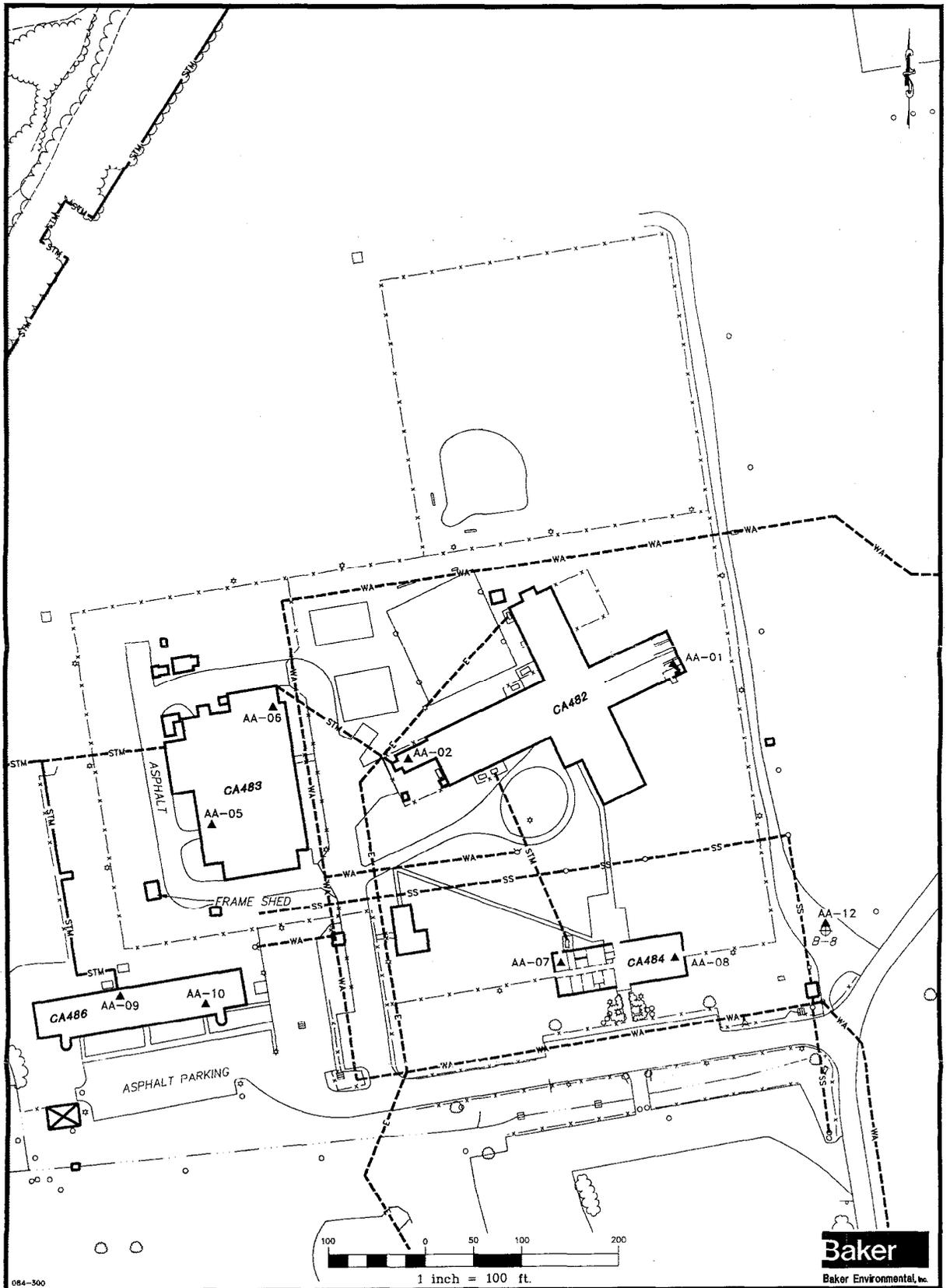
### **3.3 Air Sampling Locations: Camp Allen Elementary School (Area B)**

The Camp Allen Elementary School and surrounding area are depicted in Figure 3-4. For the air sampling program, the school has been delineated into the following sections: Section A consists of ten classrooms/lavatories, the mechanical/equipment rooms, the dining/kitchen area, an auditorium and administrative offices; Sections B and C each consist of ten

TABLE 3-1

AIR SAMPLING LOCATIONS: BRIG FACILITY (AREA A)

Building Number	Level	Sample Designation	Comments (Rationale)
CA 482	First	AA-01	Lavatory area near dormitory 1A. Utility network and potential air emission pathway.
CA 482	First	AA-02	Near utility network (steam)/kitchen area. Potential air emission pathway and chronic exposure conditions.
CA 482	Second	AA-03	Lavatory area near dormitory #3/stairwell #2. Utility network potential air emission pathway.
CA 482	Third	AA-04	Lavatory/stairwell #1 near dormitory #6. Utility network potential air emission pathway.
CA 483	First	AA-05	Maintenance area. Proximity of building to determined "Hot Spot", potential air emission pathway and chronic exposure conditions.
CA 483	First	AA-06	Maintenance area. Proximity of building to determined "Hot Spot", network of steam line, potential air emission pathway, and chronic exposure conditions.
CA 484	First	AA-07	Northwest corner of building. Proximity of building to determined "Hot Spot", potential air emission pathway and chronic exposure conditions.
CA 484	First	AA-08	Eastern corridor of building, proximity of building to determined "Hot Spot", potential air emission pathway and chronic exposure conditions.
CA 486	First	AA-09	Near steam network. Proximity of building to "Hot Spot", potential air emission pathway, and chronic exposure conditions.
CA 486	First	AA-10	Near lavatory. Utility network and potential air emission pathway
CA 486	Second	AA-11	Near lavatory/stairwell. Utility network and potential air emission pathway.
B-8	Ground	AA-12	Gas monitoring station. Potential emission source.



3-4

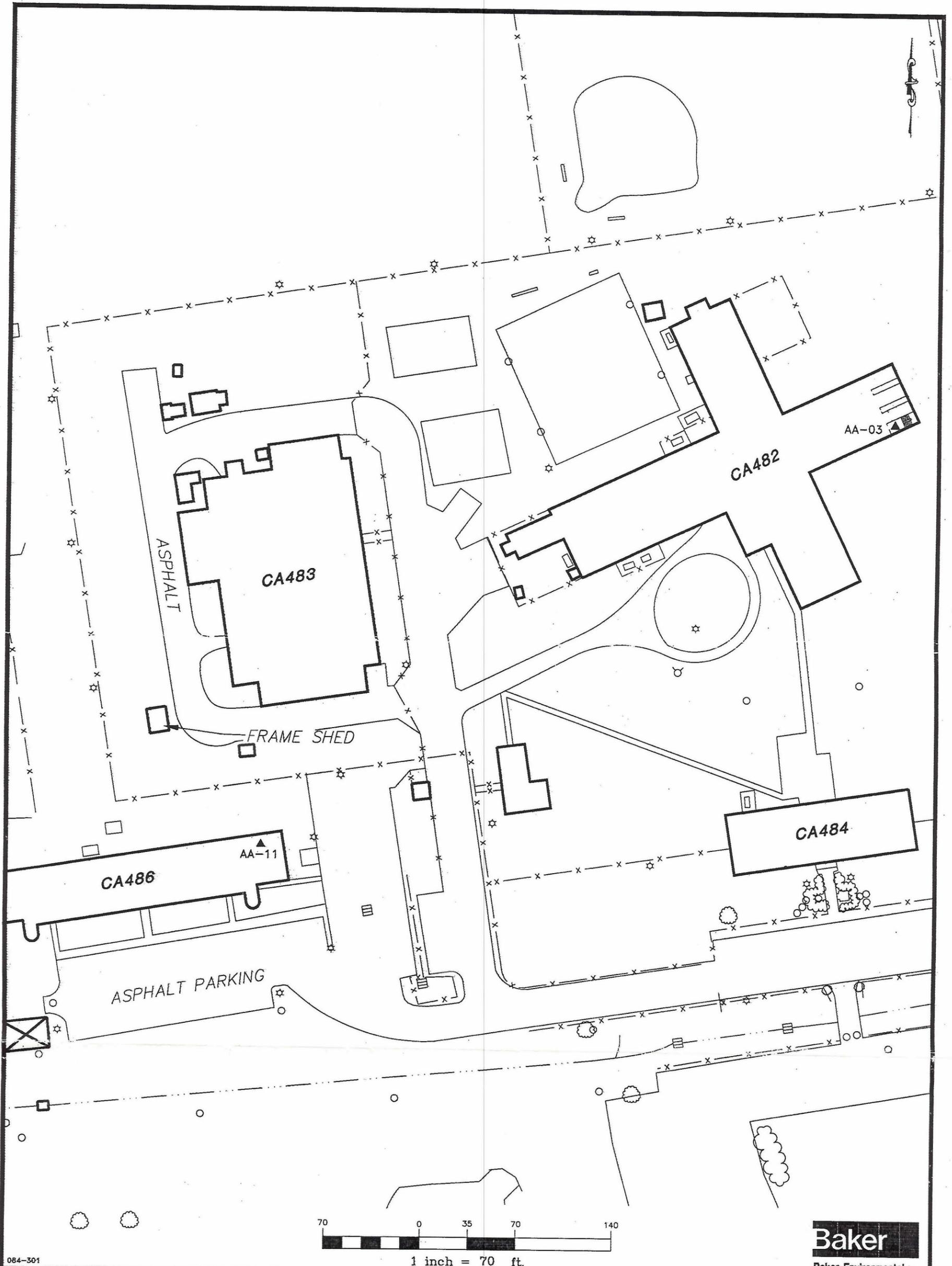
084-300

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LEGEND	
$\oplus$ B-8	GAS WELL
▲ AA-01	LOCATION OF SUMMA CANISTER
—SS—	APPROXIMATE LOCATION OF SANITARY SEWER
—S—	APPROXIMATE LOCATION OF STORM SEWER
—STM—	APPROXIMATE LOCATION OF STEAM LINE
—E—	APPROXIMATE LOCATION OF ELECTRIC LINE
—WA—	APPROXIMATE LOCATION OF WATER LINE

SOURCE: MILLER-STEPHENSON & ASSOC. JUNE 1992

FIGURE 3-1  
AIR SAMPLING LOCATIONS (FIRST FLOOR)  
AND UTILITY DETAIL  
CAMP ALLEN BRIG FACILITY (AREA A)  
NORFOLK NAVAL BASE  
NORFOLK, VIRGINIA



3-5

084-301

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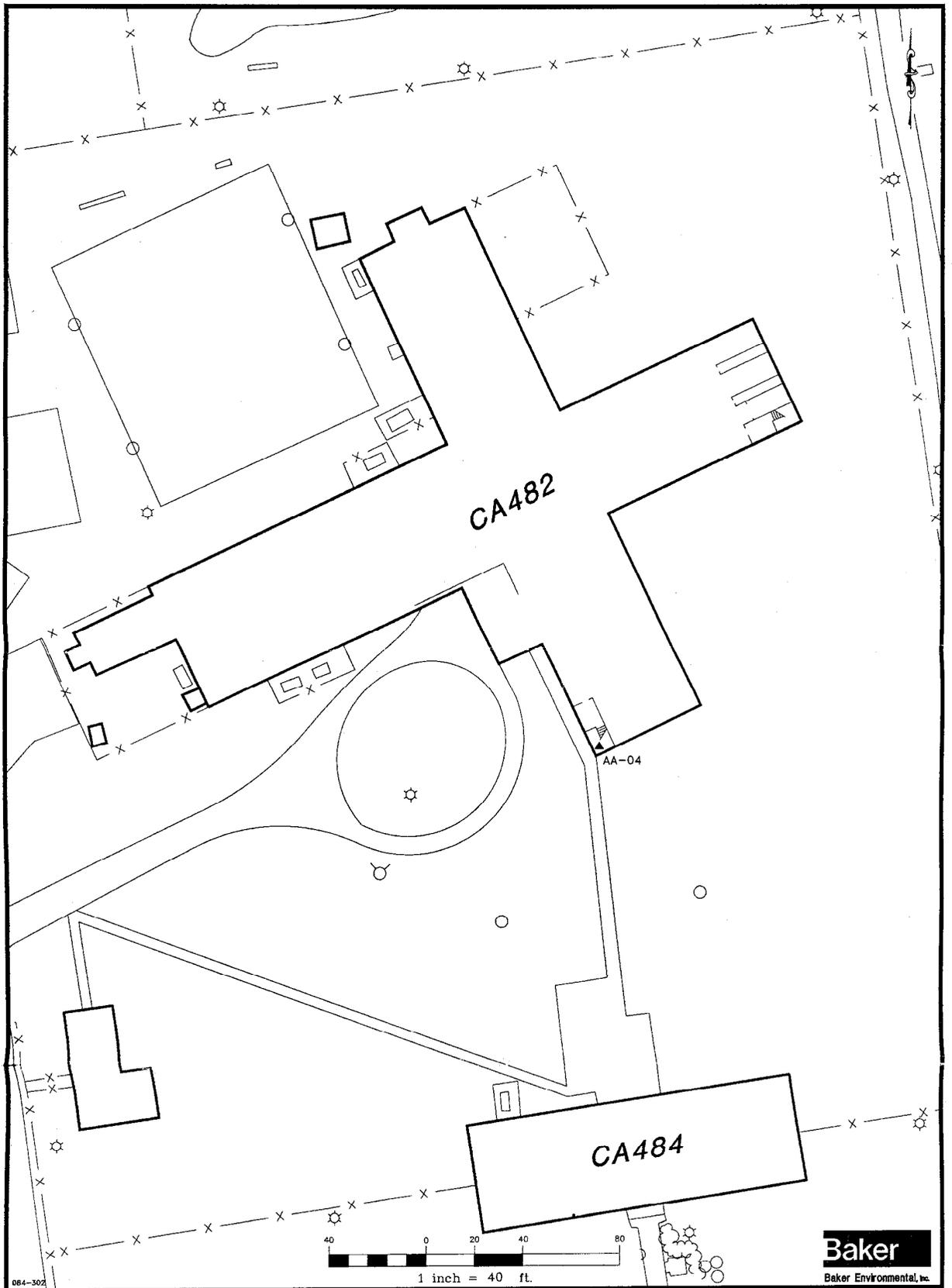
**LEGEND**

AA-03  
▲ LOCATION OF SUMMA CANISTER

**FIGURE 3-2**  
AIR SAMPLING LOCATIONS (SECOND FLOOR)  
CAMP ALLEN BRIG FACILITY (AREA A)

SOURCE: MILLER-STEPHENSON & ASSOC. JUNE 1992

NORFOLK NAVAL BASE  
NORFOLK, VIRGINIA



3-6

084-302

**LEGEND**

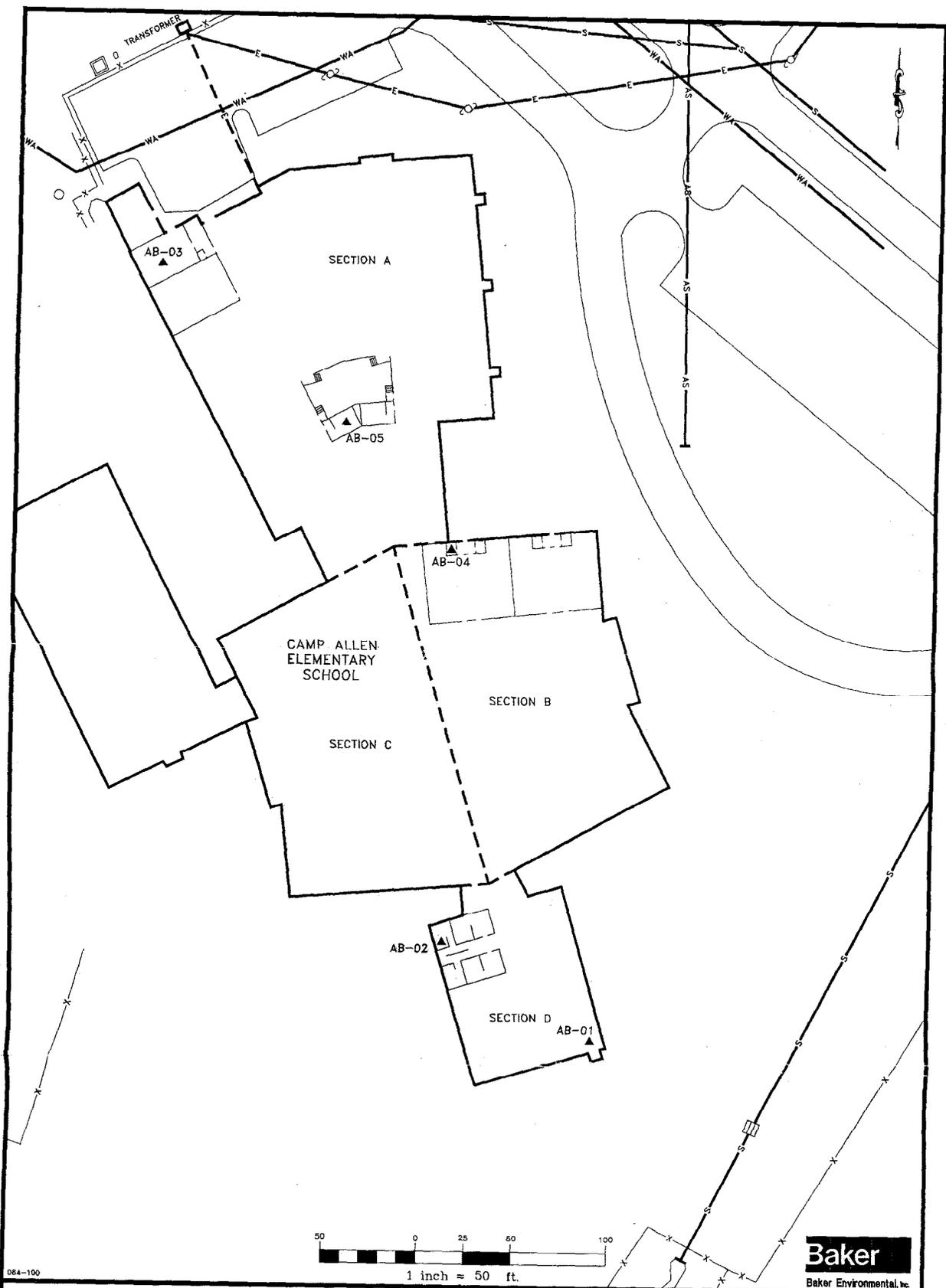
AA-04    ▲    LOCATION OF SUMMA CANISTER

**FIGURE 3-3**  
**AIR SAMPLING LOCATIONS (THIRD FLOOR)**  
**CAMP ALLEN BRIG FACILITY (AREA A)**

NORFOLK NAVAL BASE  
 NORFOLK, VIRGINIA

SOURCE: MILLER-STEPHENSON & ASSOC. JUNE 1992





084-100

**LEGEND**

- ▲ AB-01 LOCATION OF SUMMA CANISTER
- S— APPROXIMATE LOCATION OF ACTIVE STORM SEWER
- AS— APPROXIMATE LOCATION OF ABANDONED STORM SEWER
- E— APPROXIMATE LOCATION OF ELECTRIC LINE (ABOVE/BELOW)
- WA— APPROXIMATE LOCATION OF WATER LINE

**FIGURE 3-4**  
**AIR SAMPLING LOCATIONS AND UTILITY DETAIL**  
**CAMP ALLEN ELEMENTARY SCHOOL (AREA B)**

NORFOLK NAVAL BASE  
 NORFOLK, VIRGINIA

SOURCE: LANTDIV, OCT. 1991

classrooms/lavatories and general work stations; Section D consists of the gymnasium, two lavatories, storage rooms and offices. The building area is approximately 65,000 square feet and has a volume of 951,630 cubic feet on one floor level.

Based on previous investigation results, volatile organic contaminants in shallow groundwater appear to be migrating in a southerly direction along both the underground utility conduit for the storm sewer system located adjacent to the eastern portion of the school, and an underground water pipeline which trends southeast from the salvage yard area. Proposed sample locations have been selected based on a rating of the following factors: (1) detections of volatile organics in the shallow groundwater; (2) shallow groundwater flow direction; (3) underground conduits present in the surrounding area; and (4) underground utility "hook up" areas at the school facility. Figure 3-4 presents air sampling locations for the Camp Allen Elementary School. Table 3-2 provides a summary of sample locations and rationale for the Camp Allen Elementary School. No air sampling locations have been implemented in Section C of the school based on the suspected migration of contaminants and the previously listed factors.

### 3.4 Ambient Air Sampling Locations

Air contaminants may pose a threat to the health of the public downwind of the site via established "routes of entry". This threat is heightened when waste excavation/removal (construction) activities occur. The routes of entry of concern during such construction activities would primarily be inhalation and absorption; secondarily, the concern would be skin/eye contact and ingestion. Wind direction and speed are the primary factors governing transport of air contaminants (gases). Sudden, unexpected shifts in wind direction are of particular concern because they can cause exposure to site workers and the public in areas considered to be safe during non-construction periods. Appendix A contains wind rose data which was originally prepared for the Federal Aviation Administration (FAA) for aviation-related applications. The associated graphs depict wind direction versus wind speed for all weather conditions in the Norfolk, Virginia airport area. To supplement the rationale for the outdoor ambient air sampling, and placement of the SUMMA canisters, the wind rose data was evaluated. During the month of January, average climatic conditions for Norfolk, Virginia are as follows:

- Temperature      41°F
- Wind speed        11 knots
- Wind direction    Primarily from the north-northeast

TABLE 3-2

AIR SAMPLING LOCATIONS: CAMP ALLEN ELEMENTARY SCHOOL (AREA B)

Building Number	Section	Sample Designation	Comments (Rationale)
School	Section D	AB-01	Southwest corner of gymnasium. Groundwater flow and proximity of building to potential air emission pathway.
School	Section D	AB-02	Gymnasium showers. Utility network and potential air emission pathway.
School	Section A	AB-03	Maintenance area. Utility network, air emission pathway, and potential exposure conditions.
School	Section B	AB-04	Classroom #3. Groundwater flow, air emission pathway and potential exposure conditions.
School	Section A	AB-05	Lavatory adjacent to school stage. Utility network, groundwater flow, and air emission pathway.

For the purpose of this investigation, all ceiling and visibility conditions were assessed (see Appendix A). Wind direction versus wind speed tables depicting daytime distributions for six ceiling visibility classes were also reviewed based on the time and duration of the proposed air sampling program.

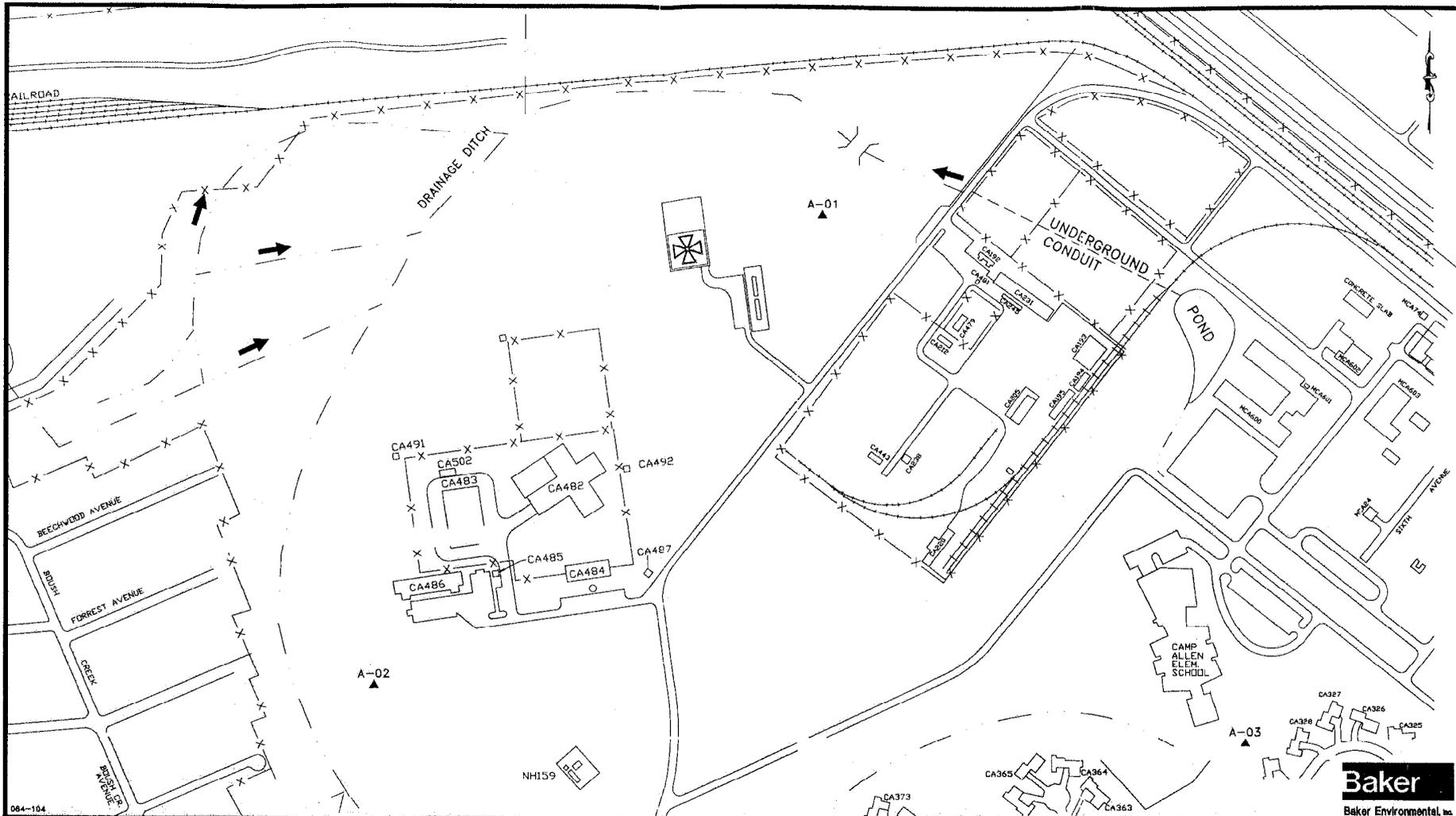
Air releases may occur from volatilization of contaminated soils from covered landfills (with and without internal gas generation). Temperature, atmospheric pressure and ground cover influence the rate of air releases. With increasing temperatures, decreasing pressures, and permeable ground covers, the rate of volatilization of compounds tends to increase.

Ambient air quality is also dependent on background conditions which include potential off-site (unrelated) sources. As the Camp Allen site is located adjacent to the Naval Air Station and in close proximity to a major highway, ambient air sampling locations must also address potential upwind/downwind variations possibly due to off-site sources. Therefore, ambient air sampling locations may be repositioned or increased based on site concerns and present wind directions.

Three ambient air locations have been selected outside of the study area based on the previously stated factors, historical information and recent groundwater analytical results. It is anticipated that the three locations will represent background conditions as well as potential source indicators. Figure 3-5 presents ambient air sampling locations outside of the Brig Facility. Table 3-3 provides a summary and rationale for ambient air sampling locations.

### **3.5 Analytical Methodology**

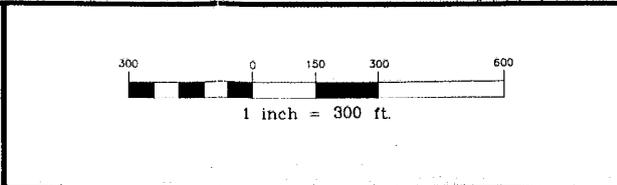
Samples collected during this investigation will be analyzed via U.S. Environmental Protection Agency (USEPA) Compendium Method TO-14 which is applicable for the determination of a wide variety of volatile organic compounds and was specifically established for the collection of whole air samples in SUMMA electropolished stainless steel canisters. Analysis by gas chromatography/mass spectrometry (GC/MS) will provide detection limits of 0.2 parts per billion (ppb) for most volatile compounds. Section 5.0 provides a detailed discussion on Method TO-14.



**LEGEND**

A-01 LOCATION OF SUMMA CANISTER

SOURCE: LANTDIV, OCT. 1991



**FIGURE 3-5**  
**AMBIENT AIR SAMPLING LOCATIONS**  
**CAMP ALLEN LANDFILL**  
**(AREAS A AND B)**

NORFOLK NAVAL BASE  
 NORFOLK, VIRGINIA

**TABLE 3-3**

**AMBIENT AIR SAMPLING LOCATIONS: CAMP ALLEN LANDFILL (AREAS A & B)**

Ambient Air Location	Level	Sample Designation	Comments (Rationale)
NE CA 486	Ground	A-01	Background Sample Point. Upwind of predicted wind direction.
SW CA 486	Ground	A-02	Based on wind direction, wind rose, historical information and analytical results. Downwind of predicted wind direction.
SCAES	Ground	A-03	Based on wind direction, wind rose, historical information and analytical results. Lateral (east) of predicted wind direction and downwind.

\*NOTE: Wind direction and speed during sampling activities may effect placement of all Summa Canisters in ambient air locations.

## 4.0 SAMPLING AND ANALYSIS PLAN

### 4.1 Purpose and Method

The SUMMA Canister sampling system (USEPA Method TO-14) is a very simple and quick method for sampling volatile organic compounds (VOCs) in ambient air. The method is based on samples of whole air collected in SUMMA passivated stainless steel canisters. The VOCs are subsequently separated by gas chromatography and measured by a mass selective detector or multidetector technique.

The SUMMA canister technique can collect sub-ambient, ambient and positive pressure samples for storage and subsequent analysis using one of three procedures. The procedure selected for the Camp Allen Landfill Site involves using an evacuated canister with a critical orifice to collect a sample at sub-atmospheric pressure at a preset flow rate over a predetermined period of time. This passive sampling mode uses an initially evacuated (sub-atmospheric pressure) canister with a mass flow controller to regulate flow. A sample of ambient air is drawn through a sampling train comprised of components that regulate the rate and duration of the sample flow rate. This technique may be utilized to collect grab samples over a period of 15 minutes to 8 hours using a six liter canister. For this sampling event samples will be collected over an 8-hour period at each of the 20 locations on three consecutive days. Appendix B provides a detailed description of procedures for sampling and analysis of volatile organic compounds in ambient air.

It should be noted that method TO-14 utilizes three different sample analysis systems (GC/MS SCAN, GC/MS SIM, and GC Multidetector). The Camp Allen Air Sampling Program will utilize the GC/MS SCAN analytical system for sample analysis. The GC/MS is set in the scan mode where the MS screens the sample for identification and quantitation of volatile organic species.

### 4.2 Sample Numbering

In order to identify and accurately track the various samples to be collected during this investigation, a numbering system has been developed. A two letter designation will be used to denote sampling media and area of the site (i.e., AA for air samples in Area A and AB for air samples in Area B). Ambient air samples collected outside of the facilities will be denoted with a single letter designation (A). Two numeric digits will succeed the letter designation

denoting sample location points (i.e., 01 denotes sample location number one, 02 designates sample location number two etc.). In addition, a single letter designation for each day of sampling will follow the two digit number specifying round one, two or three (i.e., AA-01A denotes round one, AA-01B denotes round two and AA-01C denotes round three). All air sampling numbers and respective data will be represented in field logbooks as well as a Canister Sampling Field Data Sheet (see Appendix C).

#### 4.3 Quality Assurance/Quality Control (QA\QC)

One field blank per sampling event will be provided to accommodate quality assurance requirements. The results of the quality control samples (blanks) will be evaluated for contamination. This information will be utilized to qualify the sample results in accordance with the data quality objectives. The analytical lab will follow EPA guidelines appropriate for the TO-14 Method.

Baker personnel will provide Camp Allen Elementary School officials and Navy Brig personnel with briefings regarding the Camp Allen Air Sampling Program. On-site quality control measures will be established at this time to supplement sample integrity. Security measures will also be instituted in areas deemed necessary. In addition, during sampling events Baker personnel will be conducting hourly observations at each canister to ensure sample integrity. All documentation will be reported in field logbooks and/or Canister Sampling Data Sheets.

## 5.0 QUALITY ASSURANCE PROJECT PLAN

### 5.1 Introduction and Scope

This addendum to the Quality Assurance Project Plan (QAPP) for the Remedial Investigation of the Camp Allen Landfill addresses the quality assurance and quality control (QA/QC) procedures that will be utilized for sample collection and analysis of ambient air samples. The air samples will be collected to determine the potential exposure of personnel, located downwind and downgradient of the landfill, to hazardous substances. The facilities to be sampled are the Brig and nearby elementary school. Samples will be collected at 12 locations in and around the Brig Facility, five locations in and around the school building and at three ambient air locations in the study area. The samples will be collected at each location on three consecutive days with a travel blank included for analysis with each days' samples.

The air samples will be collected using six liter SUMMA passivated stainless steel canisters at sub-atmospheric pressure and analyzed for volatile organic compounds. The VOCs are separated by gas chromatography and identified by mass spectrometry.

### 5.2 Quality Assurance Objectives for Data Measurement

The project quality assurance objectives are as described in the Quality Assurance Project Plan (QAPP), Remedial Investigation, Camp Allen Landfill, United States Naval Base, Norfolk, Virginia, April 23, 1992. The data quality objectives are expressed in terms of precision, accuracy, and completeness. These terms are defined in the project QAPP as referenced above.

The accuracy will be determined by injecting VOC standards from an audit cylinder (standard of analytes) into a sampler and then analyzing for the components in the canister. The percent relative accuracy is calculated as follows:

$$\% \text{ Relative Accuracy} = \frac{X - Y}{X} \times 100$$

Where: Y = concentration of the targeted compound recovered from a sample  
X = concentration of the targeted analyte in the audit cylinder

The relative accuracy shall be between 90 and 110 percent for all targeted VOCs. If the relative accuracy is not within these limits, the procedure should be repeated with a cleaned sampler:

The precision will be determined by the calculated relative percent difference (RPD) between laboratory duplicate analyses. The precision should be less than 25 percent. The RPD is calculated as follows:

$$RPD = \frac{(C1 - C2)}{\left(\frac{C1 + C2}{2}\right)} \times 100$$

Where: C1 = larger of the two observed values  
C2 = smaller of the two observed values

Completeness is the percentage of all measurements judged to be valid. The objectives for completeness are to obtain 90 percent valid data.

The data quality objective for this phase of the project is Level E, which is defined in Section 1.3.3 of "Sampling and Chemical Analysis Quality Assurance Requirements for the Navy Installation Restoration Program," NEESA 20.2-047B, June 1988. Level E is used for analysis of nonstandard sample matrices, such as air, biota, and pure product or waste. Level E requires that the analytical methods used be accepted EPA methods, if available. Therefore, the sampling and analytical methods selected for this project are EPA methods which have been used extensively within EPA programs (EPA's Toxics Air Monitoring Stations, Urban Air Toxic Pollutant Program, and the non-methane organic compound sampling and analysis program).

For Level E, a blank/spike control shall be analyzed with each batch of samples and the results shall be plotted on control charts. Initial and continuing calibrations and method blanks are also required.

In Level E, the only information to be provided by the laboratory is the sample data, method blank data, and the control charts plotted from the blank/spike data.

### 5.3 Sampling Procedure

The details of the procedure to be used for ambient air sampling are presented in Section 4.0. The SUMMA passivated canisters, prepared for sub-atmospheric sampling, will be provided by the analytical laboratory subcontractor. The interior surfaces of the canisters are treated by the SUMMA passivation process, in which a pure chrome-nickel oxide is formed on the surface. This type of vessel has been demonstrated to provide a stable sample storage surface for many specific organic compounds. The canisters and associated field sampling equipment will be cleaned and certified by the laboratory subcontractor.

### 5.4 Analytical Procedures

The samples that will be collected during this investigation will be analyzed for VOCs by EPA Method TO-14 "Determination of Volatile Organic Compounds in Ambient Air Using SUMMA® Passivated Canister Sampling and Gas Chromatographic Analysis," May 1988. The method is based on the collection of ambient air samples by sub-atmospheric pressure sampling with passivated 6-liter stainless steel canisters. Integrated samples will be obtained by continuous sampling over an eight-hour period. The VOCs are analyzed by separation of targeted analytes by capillary gas chromatography and measured by mass spectrometry. This method is applicable to specific VOCs that have been tested and determined to be stable when stored in sub-atmospheric pressure canisters. The mass spectrometer will be operated in the scan mode over the mass range of 18 to 250 amu. The target analytes that will be measured are listed in Table 5-1 with their corresponding detection limits. These compounds have been successfully analyzed at the low parts per billion by volume (ppbv) level.

The laboratory that has been subcontracted to perform the analyses is International Technology Analytical Services - Cincinnati, Ohio. NEESA has not approved any laboratories for the analysis of air samples at this time. The selection of the laboratory subcontractor was made on the basis of the subcontractor's qualifications, personnel experience in the analysis of air samples, the equipment available at the laboratory, and the cost effectiveness of the analyses as determined by a competitive bidding process. In addition, a review of the laboratory's Quality Assurance Plan will be made prior to the start of the analyses. NEESA has been notified of the laboratory selected for this project.

TABLE 5-1

## TARGET ANALYTES AND DETECTION LIMITS

Compound	Approximate Detection Limit (ppb)	Compound	Approximate Detection Limit (ppb)
Dichlorodifluoromethane	0.2	1,1,2-Trichloroethane	0.2
Chloromethane	0.2	Tetrachloroethene	0.2
Vinyl chloride	0.2	1,2-Dibromomethane	0.2
Bromomethane	0.2	Chlorobenzene	0.2
Chloroethane	0.2	Ethylbenzene	0.2
1,1-Dichloroethene	0.2	m- and/or p-Xylene	0.2
Freon 113*	0.2	o-Xylene	0.2
Methylene Chloride	0.2	Styrene	0.2
1,1-Dichloroethane	0.2	1,1,2,2-Tetrachloroethane	0.2
c-1,2-Dichloroethene	0.2	1,3-Dichlorobenzene	0.1
Chloroform	0.2	1,4-Dichlorobenzene	0.1
1,1,1-Trichloroethane	0.2	Benzyl Chloride	0.2
Carbon tetrachloride	0.2	1,2-Dichlorobenzene	0.1
Benzene	0.2	Freon 114**	0.2
1,2-Dichloroethane	0.2	Trichlorofluoromethane	0.2
Trichloroethene	0.2	1,3,5-Trimethylbenzene	0.2
1,2-Dichloropropane	0.2	1,2,4-Trimethylbenzene	0.2
c-1,3-Dichloropropene	0.2	1,2,4-Trichlorobenzene	0.1
Toluene	0.2	Hexachlorobutadiene	0.1
t-1,3-Dichloropropene	0.2		

ppb = parts per billion

\* Freon 113 = 1,1,2-Trichloro-1,2,2-trifluoroethane

\*\* Freon 114 = 1,2-Dichloro-1,1,2,2-Tetrafluoroethane

## **5.5 Quality Assurance and GC/MS/Scan System Performance Criteria**

The sampling and analysis quality assurance procedures are described in the EPA Compendium Method TO-14. The required QA/QC elements are outlined below.

### **5.5.1 Sampling System**

- Pre- and post-sampling measurements are made with a certified mass flow controller for flow verification of the sampling system.
- All canisters shall be pressure tested over a 24-hour period.
- All canisters and field sampling systems shall be certified clean (containing less than 0.2 ppbv) through a humid zero air testing program.
- All field sampling systems must pass a humid calibration gas certification at concentration levels expected in the field with a minimum 90 percent recovery.

### **5.5.2 Gas Chromatograph - Mass Spectrometer - SCAN System Performance Criteria**

- The GC-MS analytical system shall be certified clean (less than 0.2 ppbv of target VOCs).
- The GC-MS system must be tuned daily with 4-bromofluorobenzene (BFB) and the system must meet the key ions and ion abundance criteria (10%) outlined in Table 5-2.
- An initial multi-point humid static calibration and a daily calibration of the GC/MS system is required.

## **5.6 Calibration Procedures and Frequency**

The laboratory's procedures for calibration and related quality control measures are to be in accordance with the requirements stated in the EPA Compendium Method TO-14. The general calibration procedures are discussed in the QAPP for the Camp Allen RI/FS.

**TABLE 5-2**  
**4-BROMOFLUOROBENZENE KEY IONS AND ION**  
**ABUNDANCE CRITERIA**

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

The GC/MS calibration procedures as identified in the analytical method include an initial calibration. This is a multi-point dynamic calibration (five concentration levels plus humid zero air) performed on the GC/MS system, prior to sample analysis. The calibration standards must be NIST traceable standards or NIST/EPA certified reference materials. After the GC/MS system is calibrated at five concentration levels, a second humid zero air sample is used to verify that the system is clean.

The GC/MS system is also calibrated daily (every 12 hours before sample analysis) with a one-point calibration (check standard). After the single point calibration, the analytical system is tested with humid zero gas to insure that the GC/MS system is within specifications (less than 0.2 ppbv of target organics).

The percent relative standard deviation of the response factors for the initial calibration must be less than 25 percent. The response for the continuing calibration standard must be within  $\pm 25$  percent for the target analytes or a new curve must be developed. The mass spectrometer tune must be verified by performing an analysis of BFB prior to analyzing each check standard.

## **5.7 Internal Quality Control Checks**

The analytical laboratory shall perform several internal quality control (QC) checks during the period of time the samples are analyzed. These QC procedures and frequencies are discussed in this section and summarized in Table 5-3.

### **5.7.1 Calibration**

Calibration standards for VOCs analyses are prepared from neat compounds and the accuracy is verified by the lab or supplier. An internal audit sample for canister analysis shall be analyzed weekly to verify the prepared standards.

### **5.7.2 Method Blanks**

A certified canister is filled with humid air and analyzed in the same manner as a sample.

**TABLE 5-3**  
**FREQUENCIES OF INTERNAL LABORATORY**  
**QUALITY CONTROL CHECKS**

QC Test	Frequency	Acceptance Criteria*
Initial calibration	When CCC fails	± 25%
Continuing calibration	Daily	± 25%
Method blanks	10%	<DL
Precision (duplicates)	10%	± 30%
Audit sample (spike)	5%	± 30%
Surrogates	All samples	± 30%

\* From the Laboratory's Quality Assurance Project Plan (Revision 10, August 1992)

### **5.7.3 Accuracy**

Surrogates are added to each sample for analysis and the percent recoveries are calculated.

### **5.7.4 Precision**

Duplicate analyses are used to determine precision.

### **5.7.5 Pressure Gauges**

NIST certified, 0.2 pounds per square inch (psi) per division, pressure gauges will be used to read the final pressure in the sampling canisters. These gauges will be certified at intervals of 6 months or less. These gauges are used to accurately quantify collected sample volumes.

## 6.0 HEALTH AND SAFETY PLAN

The Health and Safety Plan for CTO-0084 contains detailed information regarding the procedures which will be followed during field activities at the Camp Allen Landfill Areas A and B. That plan, referred to as the CTO-0084 HASP, should be used in conjunction with this Health and Safety Plan Addendum. For reference, the CTO-0084 HASP is included as an attachment to the Final Work Plan dated April 23, 1992.

### 6.1 Site Entry Objectives

The objectives of the initial entry to this area are to characterize the degree, type, and extent of ambient air contamination. This investigation will include the following on-site activities: initial scoping, staging of SUMMA passivated canisters, periodic data collection at specified locations, and dismantling of air sampling equipment.

### 6.2 Site Organization and Coordination

The following personnel are designated to carry out the stated job functions on-site. Note: One person may carry out more than one job function. However, when such collateral duties appear to require simultaneous attention, any health and safety activities will take immediate precedence over any other duty(s).

- Baker Project Manager: Mr. Thomas Artman
- Health and Safety Officer: Ms. Barbara Cummings
- Environmental Scientist: Mr. Kenneth Martin
- Federal Agency Representatives
- LANTDIV EIC: Primary: Mr. Kenneth Walker (804) 445-4385  
Alternate: Ms. Susan Hauser (804) 445-8856
- Activity Base Representatives
  - Activity Coordinator: Ms. Cheryl Barnett (804) 444-3009
  - Alternate: Mr. Dave Forsythe (804) 444-3009
- Contractor(s)
  - Analytical Laboratory: International Technology (IT) (513) 782-4600

### **6.3 Site Control**

The procedures which will be followed for site control are described in the CTO-0084 HASP, Section 5.0.

### **6.4 Hazard Evaluation**

The hazard evaluations for this site are contained in the CTO-0084 HASP, Section 6.0.

### **6.5 Emergency Services Location**

An emergency services route map depicting the location of the local hospital will be posted and available for site personnel. Personnel will be informed of the map and direction to the hospital.

Directions to DePaul General Hospital follow (Refer to Figure 6-1):

1. From Area A or B proceed west on Ingersol Street until arriving at a "T", then turn right and a quick left onto Helmick Street.
2. Proceed on Helmick Street until intersecting with Hampton Boulevard (Route 337), then turn left and proceed south on Route 337 until intersecting with Little Creek Road.
3. Turn left onto Little Creek Road and proceed east for approximately 2-1/4 miles until intersecting with Granby Street, then turn right (south).
4. Continue on Granby Street until intersecting with Kingsley Lane (approximately 1-1/2 miles) and DePaul General Hospital will be on right-hand side.

Site Address: Baker Site Trailer  
B Street (Adjacent to Salvage Yard)  
Camp Allen  
Norfolk, Virginia 23511

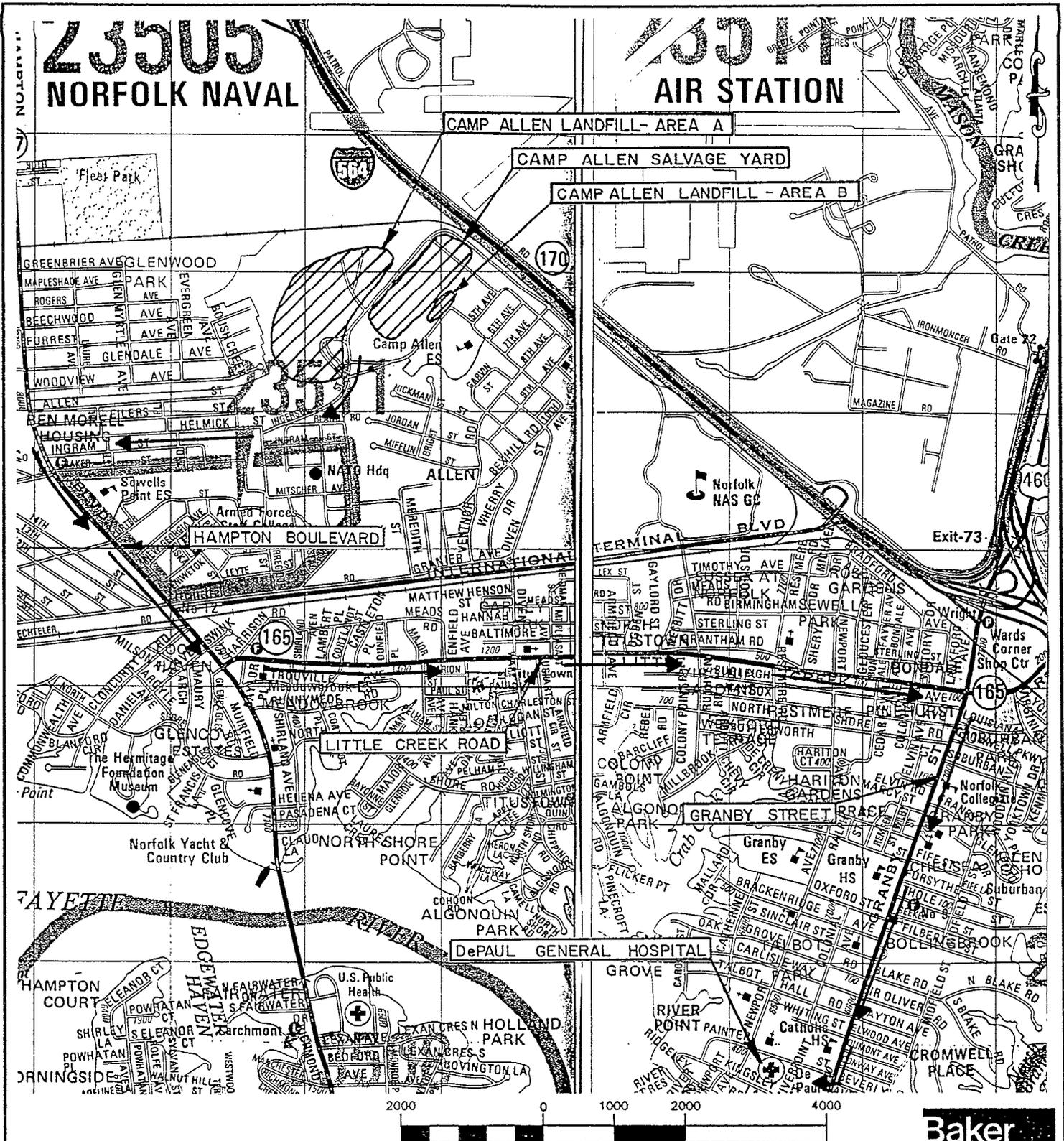


FIGURE 6-1  
 EMERGENCY HOSPITAL ROUTE  
 CAMP ALLEN LANDFILL  
 AREAS A AND B  
 NORFOLK NAVAL BASE  
 NORFOLK, VIRGINIA

SOURCE: ADC OF ALEXANDRIA, INC., 1990

1 inch = 2000ft.

**Baker**  
 Baker Environmental, Inc.

## REFERENCES

Malcolm Pirnie, Inc., (June 1984), NACIP Program Confirmation Study-Site Suitability Assessment, Proposed Brig Expansion (P-977), Naval Station, Norfolk, VA.

U.S. Department of Commerce. National Oceanic and Atmospheric Administration. National Environmental Satellite Data and Information Service, National Climatic Data Center Federal Building. November 1992.

U.S. Environmental Protection Agency. Region III Technical Assistance Team, Standard Operating Procedures, Air Sampling Using SUMMA Canister Samplers. February 1991.

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U.S. Environmental Protection Agency. Air/Superfund National Technical Guidance Study Series, Volume II - Estimation of Baseline Air Emissions at Superfund Sites - Interim Final. EPA-450/1-89002. January 1989.

**APPENDIX A**  
**USE OF ANNUAL WIND ROSE FOR**  
**CLIMATIC APPLICATIONS**

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UNITED STATES DEPARTMENT OF COMMERCE  
National Oceanic and Atmospheric Administration  
NATIONAL ENVIRONMENTAL SATELLITE, DATA,  
AND INFORMATION SERVICE  
NATIONAL CLIMATIC DATA CENTER  
FEDERAL BUILDING  
ASHEVILLE, NORTH CAROLINA 28801

### Use of Annual Wind Rose for Climatic Applications

The attached wind rose was originally prepared for the FAA for aviation related applications. The graph depicts wind direction versus wind speed during all weather conditions at a particular airport site. Extreme caution must be taken if this graph is used for other applications for the following reasons:

1. The graph is an annual distribution and does not take into account seasonal or monthly variations in wind speed and direction.
2. The graph does not take into account daily variations in wind speed and direction. For example, at most coastal locations a land-sea breeze is common during fair weather. During the day at Daytona Beach, Florida, 50.2 percent of all winds are easterly, while 35.3 percent are westerly. At night 33.4 percent are easterly, while 41.8 percent are westerly. Also, during the day 4.0 percent of all winds are calm compared to 12.9 percent at night.
3. The graph is for a particular airport site. Depending upon topography, the wind climate could be entirely different only a small distance away.

These problems could have serious implications if the annual graph is used for applications of short duration (hours, days, months). However, if the application is for time periods greater than a year, the attached graph is representative.



# FOREWORD

## INTRODUCTION

Summary tables and graphs have been prepared to assist the FAA in the application of establishment criteria for precision and non-precision approach and landing aids. These tables and graphs depict wind direction and speed under specified ceiling and visibility conditions. This report has been prepared by the National Climatic Center for the FAA under Interagency Agreement No. DOT-FA79WAI-057.

Climatological information for selected airports is presented in 11 volumes:

- Volume 1 - New England Region (ANE)
- Volume 2 - Eastern Region (AEA)
- Volume 3 - Southern Region (ASO)
- Volume 4 - Great Lakes Region (AGL)
- Volume 5 - Central Region (ACE)
- Volume 6 - Southwest Region (ASW)
- Volume 7 - Rocky Mountain Region (ARM)
- Volume 8 - Northwest Region (ANW)
- Volume 9 - Western Region (AWE)
- Volume 10 - Alaskan and Pacific Regions (AAL and APC)
- Volume 11 - Visibility Time Series for Key Stations

Summary tables and graphs are presented in volumes 1-10. Graphical time series of visibility measurements for key stations throughout the United States are presented in volume 11 to give the reader some insight into the climate variability that can be expected.

## ELEMENT CLASSIFICATION

Twelve tables for each station depict wind direction vs. wind speed distributions. Half the tables show daytime distributions for six ceiling-visibility classes, and half show nighttime distributions for the same six ceiling-visibility classes. A graphical depiction of the data in each table is also presented. An additional graph for each station depicts the combined day and night winds for all ceiling-visibility conditions.

Wind direction is grouped according to a 16-point compass (N, NNE, NE, ENE, E, ESE, SE, SSE, S, SSW, SW, WSW, W, WNW, NW, NNW) plus calm. The wind speed groups are 0-3, 4-12, 13-15, 16-18, 19-24, 25-31 and equal to or greater than 32 mph.

The ceiling-visibility criteria are defined as:

CLASS 1 = ceiling equal to or greater than 1,500 feet and visibility equal to or greater than 3 miles.

CLASS 2 = ceiling less than 1,500 feet and/or visibility less than 3 miles.

CLASS 3 = ceiling less than 1,500 feet and/or visibility less than 3 miles, but ceiling equal to or greater than 400 feet and visibility equal to or greater than 1 mile.

CLASS 4 = ceiling less than 400 feet and/or visibility less than 1 mile, but ceiling equal to or greater than 200 feet and visibility equal to or greater than 1/2 mile.

CLASS 5 = ceiling less than 200 feet and/or visibility less than 1/2 mile, but ceiling equal to or greater than 100 feet and visibility equal to or greater than 1/4 mile.

CLASS 6 = ceiling less than 100 feet and/or visibility less than 1/4 mile.

CLASS 7 = all ceiling and visibility conditions; sum of classes 1 and 2; also the sum of classes 1 and 3 through 6.

Time conditions are determined from the onset of civil twilight in the morning to the end of civil twilight in the evening. Civil twilight begins (ends) when the sun is six degrees below the horizon. It covers the somewhat indefinite period before sunrise and after sunset during which natural illumination usually remains sufficient for ordinary outdoor operations. For this report the morning (evening) times of beginning (ending) of civil twilight at a station are determined for the middle of a month. All data within this month are classified as either day or night according to these times.

#### DATA

The data used in the preparation of the climatological tables and graphs were extracted from the National Climatic Center's Tape Deck 1440 Hourly Surface Observations. The data contained in this deck have been digitized from the official observations recorded on station. From about 1948 through 1964 data were digitized for each hour in a day. From 1965 onward only every third hour's data were digitized. The tables and graphs in this report are based on eight observations per day (every third hour) for

the entire period of record. This was done to eliminate the data bias problems that would have been created by trying to mix the 24 observations per day prior to 1965 with the 8 observations per day after 1964.

The effect on the climatology of not using all the available data was examined for representative key stations in each region. Monthly day and night distributions were prepared for all ceiling-visibility criteria and wind speed and direction groups using 24 observations per day. A data subset was created by extracting every third observation from the hourly data set, and similar frequency distributions were prepared. The Kolmogorov-Smirnov test was applied to evaluate the hypotheses that distributions using 24 observations per day are statistically the same as distributions using 8 observations per day. The results of this procedure indicate equality

of distributions. Therefore, no significant information has been lost by basing the climatologies on 8 observations per day.

The tables and graphs are also available on microfiche, and the summarized data are available on magnetic tape. This report presents annual summaries, but monthly summaries can be generated on magnetic tape. Requests for microfiche copies of this report and for data on magnetic tape should be addressed to:

Director  
National Climatic Center  
Federal Building  
Asheville, NC 28801

The requested microfiche copies and/or tapes will be provided at cost.

## TABLE LEGEND

A = number of day (night) observations in a given ceiling-visibility class, wind speed class and direction divided by the total number of day (night) observations in ceiling-visibility classes 1, 3-6, all wind speed classes and directions times 100.

B = number of day (night) observations in a given ceiling-visibility class, wind speed class and direction divided by the total number of day (night) observations in the given ceiling-visibility class and direction times 100.

C = number of day (night) observations of a given direction divided by the total number of day (night) observations in ceiling-visibility classes 1, 3-6 times 100.

D = number of day (night) observations of a given direction divided by the total number of day (night) observations in the given ceiling-visibility class times 100.

E = number of day (night) observations in a given wind speed class divided by the total number of day (night) observations in ceiling-visibility classes 1, 3-6 times 100.

F = number of day (night) observations in a given wind speed class divided by the total number of day (night) observations in the given ceiling-visibility class times 100.

G = number of day (night) observations in the given ceiling-visibility class divided by the total number of day (night) observations in ceiling-visibility classes 1, 3-6 times 100.

100 = summation of column D = summation of row F.

H = average wind speed for the given day (night) ceiling-visibility class and direction.

I = average wind speed for the given day (night) ceiling-visibility class.

J = overall period of record; see station list for actual years of record used.

Note: Ceiling-visibility class 2 is the sum of classes 3-6.

Interagency Agreement DOT-FA79WAI-057

WIND-CEILING-VISIBILITY  
DATA  
AT SELECTED AIRPORTS

January 1981

This report has been prepared by the U. S. Department of Commerce, National Oceanic and Atmospheric Administration, Environmental Data and Information Service, National Climatic Center, Asheville, North Carolina for the Office of Aviation Policy and Plans, Federal Aviation Administration under Interagency Agreement No. DOT-FA79WAI-057. The contents of this report reflect the views of the contractor, who is responsible for the facts and accuracy of the data presented herein, and do not necessarily reflect the official views or policy of the FAA. This report does not constitute a standard, specification or regulation.



# CEILING-VISIBILITY WIND TABULATIONS

ANNUAL

STATION NAME: ORF NORFOLK, VA

PERIOD: 1948-1978

CLASS 1 / DAY

STATION NUMBER: 13737

NO. OBS.: 42689

D I R	SPEED GROUPS (MPH)														AVG WIND SPEED		
	0-3		4-12		13-15		16-18		19-24		25-31		32+			TOTAL	
	A	B	A	B	A	B	A	B	A	B	A	B	A	B		C	D
N	.3	.3	3.3	3.9	1.5	1.8	1.5	1.7	.5	.6	.1	.1	.0	.0	7.2	8.4	12.5
NNE	.2	.3	4.0	4.7	1.4	1.7	1.2	1.4	.5	.5	.1	.1	.0	.0	7.5	8.8	11.8
NE	.4	.5	5.1	6.0	1.6	1.9	1.1	1.3	.4	.5	.0	.1	.0	.0	8.7	10.2	11.2
ENE	.3	.3	2.9	3.4	.9	1.1	.6	.7	.2	.2	.0	.0	.0	.0	4.9	5.7	10.8
E	.4	.5	2.8	3.3	.6	.7	.2	.3	.0	.0	.0	.0	.0	.0	4.0	4.7	9.2
ESE	.3	.4	2.2	2.5	.4	.5	.1	.2	.0	.0	.0	.0	.0	.0	3.1	3.6	9.0
SE	.3	.3	2.7	3.2	.6	.6	.3	.3	.0	.0	.0	.0	.0	.0	3.9	4.6	9.5
SSE	.2	.3	2.1	2.5	.5	.6	.2	.3	.1	.1	.0	.0	.0	.0	3.1	3.6	9.7
S	.3	.4	3.7	4.4	1.2	1.4	.6	.8	.2	.3	.0	.1	.0	.0	6.2	7.3	10.8
SSW	.2	.3	3.1	3.7	1.5	1.7	1.2	1.4	.4	.5	.1	.1	.0	.0	6.6	7.7	12.3
SW	.4	.4	4.0	4.6	1.9	2.3	1.8	2.1	.7	.9	.1	.2	.0	.0	8.9	10.5	12.7
WSW	.2	.3	2.3	2.7	1.1	1.3	.9	1.1	.5	.6	.1	.1	.0	.0	5.3	6.2	12.7
W	.3	.3	2.5	3.0	.9	1.0	.8	1.0	.4	.5	.1	.1	.0	.0	5.0	5.9	12.2
WNW	.2	.2	1.5	1.8	.7	.8	.7	.8	.3	.4	.1	.1	.0	.0	3.5	4.1	12.7
NW	.3	.3	1.6	1.9	.6	.7	.7	.8	.3	.4	.1	.1	.0	.0	3.7	4.3	12.4
NNW	.1	.1	1.2	1.4	.7	.9	.7	.8	.4	.4	.1	.1	.0	.0	3.1	3.7	13.4
CALM	.7	.8													.7	.8	
TOTAL	5.2	6.1	45.1	52.9	16.2	19.0	12.8	15.0	5.1	6.0	.9	1.0	.1	.1	85.3	100	11.5



# CEILING-VISIBILITY WIND TABULATIONS

ANNUAL

STATION NAME: ORF NORFOLK, VA

PERIOD: 1948-1978

CLASS 1 / NIGHT

STATION NUMBER: 13737

NO. OBS.: 35004

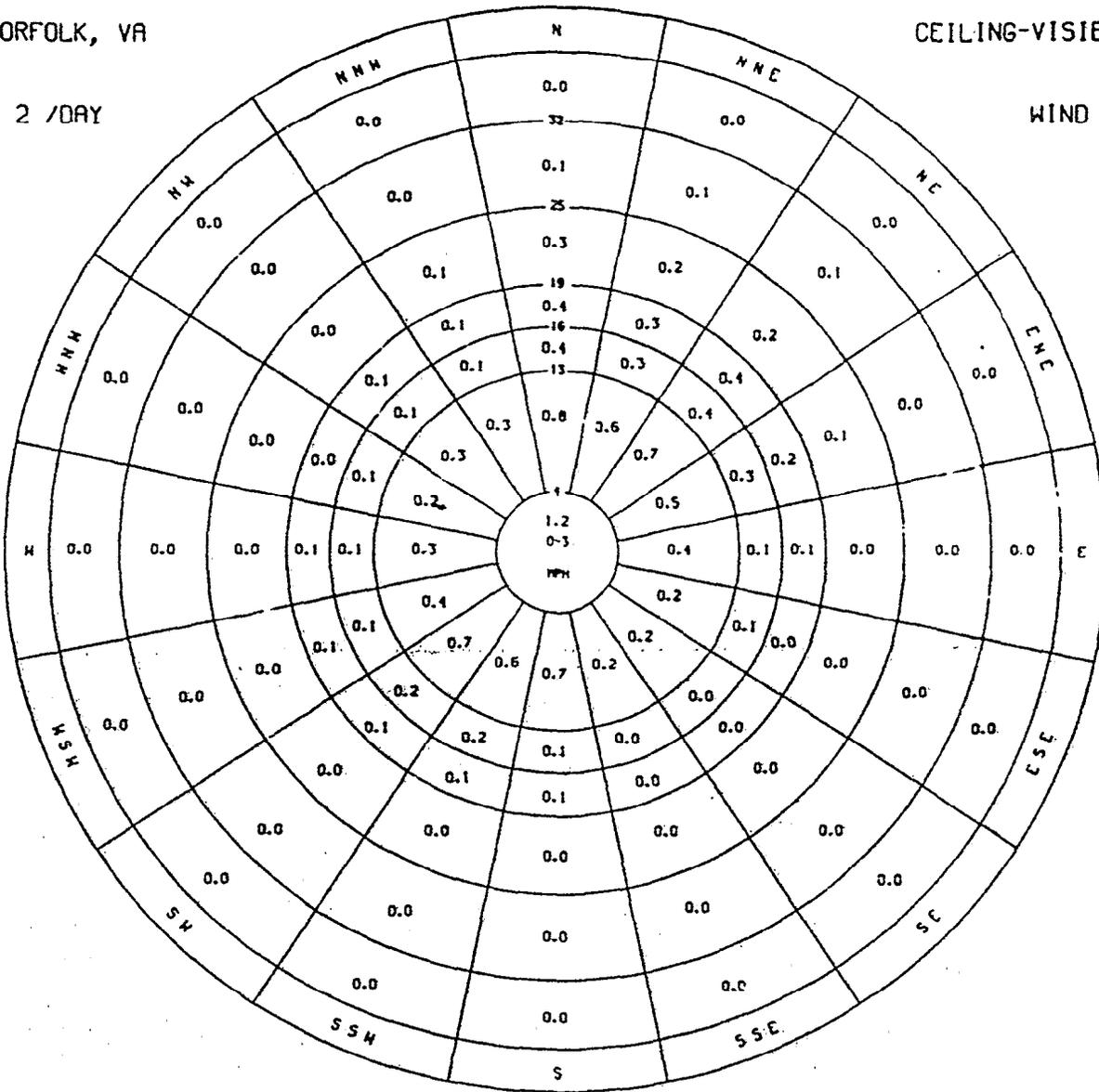
D I R	SPEED GROUPS (MPH)																AVG WIND SPEED
	0-3		4-12		13-15		16-18		19-24		25-31		32+		TOTAL		
	A	B	A	B	A	B	A	B	A	B	A	B	A	B	C	O	
N	.4	.5	2.2	2.6	1.0	1.2	1.2	1.4	.6	.7	.1	.1	.0	.0	5.5	6.4	12.7
NNE	.2	.2	1.6	1.9	.8	1.0	.9	1.0	.4	.5	.1	.1	.0	.0	4.0	4.7	12.9
NE	.5	.6	2.6	3.1	1.0	1.1	.8	.9	.3	.4	.0	.0	.0	.0	5.3	6.1	11.1
ENE	.5	.6	2.2	2.5	.3	.4	.2	.3	.1	.1	.0	.0	.0	.0	3.4	3.9	8.6
E	1.0	1.1	2.3	2.7	.1	.2	.1	.1	.0	.0	.0	.0	.0	.0	3.6	4.1	6.3
ESE	.6	.7	1.7	1.9	.1	.1	.0	.0	.0	.0	.0	.0	.0	.0	2.5	2.8	6.3
SE	1.0	1.2	2.8	3.2	.1	.2	.1	.1	.0	.0	.0	.0	.0	.0	4.0	4.6	6.4
SSE	.9	1.0	3.3	3.8	.2	.3	.1	.1	.0	.0	.0	.0	.0	.0	4.5	5.2	7.2
S	1.6	1.9	9.2	10.6	1.1	1.2	.6	.7	.2	.2	.0	.0	.0	.0	12.6	14.6	8.3
SSW	.9	1.1	6.6	7.6	1.5	1.7	.9	1.0	.2	.2	.0	.0	.0	.0	10.1	11.8	9.6
SW	1.2	1.3	6.2	7.2	1.5	1.7	.8	.9	.2	.3	.0	.1	.0	.0	9.9	11.4	9.5
WSW	.6	.7	2.5	2.9	.4	.5	.4	.4	.2	.2	.0	.1	.0	.0	4.1	4.8	9.4
W	.6	.7	2.1	2.4	.5	.6	.4	.4	.2	.2	.1	.1	.0	.0	3.8	4.5	9.7
WNW	.3	.3	1.4	1.6	.3	.4	.3	.4	.2	.2	.0	.0	.0	.0	2.6	3.0	10.7
NW	.5	.5	2.1	2.4	.6	.7	.6	.7	.3	.3	.1	.1	.0	.0	4.1	4.8	11.1
NNW	.2	.3	1.3	1.5	.6	.7	.7	.8	.4	.4	.1	.1	.0	.0	3.3	3.8	12.8
CALM	3.0	3.5													3.0	3.5	
TOTAL	14.1	16.3	50.2	58.1	10.2	11.8	8.0	9.3	3.2	3.7	.7	.8	.1	.1	86.3	100	9.2

ORF NORFOLK, VA

CLASS 2 /DAY

CEILING-VISIBILITY

WIND GRAPH



# CEILING-VISIBILITY WIND TABULATIONS

ANNUAL

STATION NAME: ORF NORFOLK, VA

PERIOD: 1948-1978

CLASS 2 / DAY

NO. OBS.: 7344

STATION NUMBER: 13737

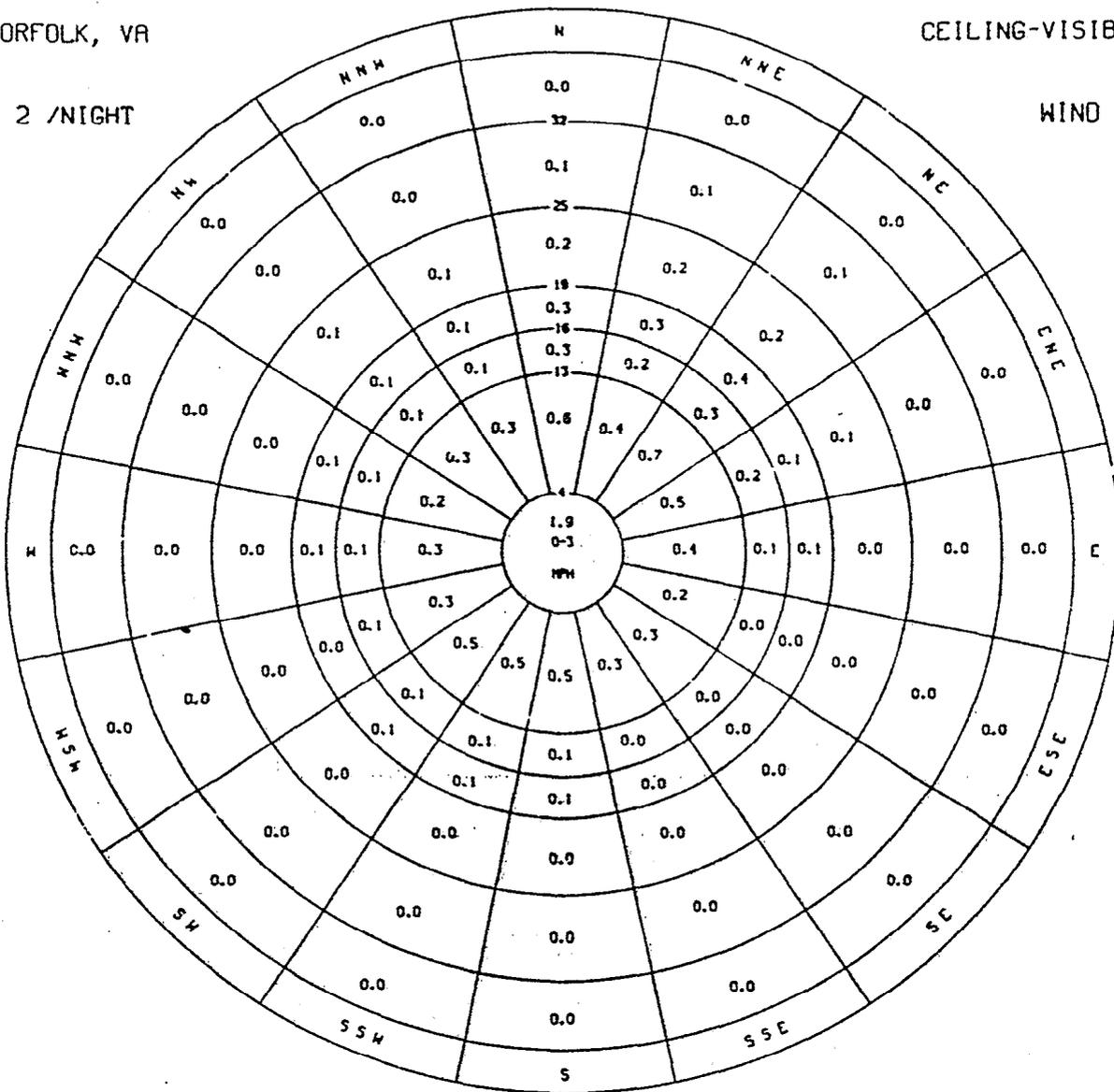
D I R	SPEED GROUPS (MPH)														AVG WIND SPEED		
	0-3		4-12		13-15		16-18		19-24		25-31		32+			TOTAL	
	A	B	A	B	A	B	A	B	A	B	A	B	A	B		C	O
N	.1	.4	.8	5.3	.4	2.4	.4	2.7	.3	1.9	.1	.5	.0	.0	1.9	13.2	13.9
NNE	.1	.4	.6	4.1	.3	1.8	.3	2.2	.2	1.3	.1	.6	.0	.0	1.5	10.4	14.1
NE	.1	.4	.7	5.1	.4	2.7	.4	3.0	.2	1.4	.1	.6	.0	.1	1.9	13.2	13.4
ENE	.0	.3	.5	3.5	.3	1.8	.2	1.6	.1	.7	.0	.2	.0	.0	.6	4.3	10.6
E	.1	.4	.4	2.5	.1	.6	.1	.5	.0	.2	.0	.0	.0	.0	.4	2.6	10.4
ESE	.0	.3	.2	1.5	.1	.4	.0	.3	.0	.2	.0	.0	.0	.0	.4	2.9	9.8
SE	.1	.5	.2	1.6	.0	.3	.0	.3	.0	.2	.0	.0	.0	.0	.4	2.7	9.8
SSE	.0	.3	.2	1.6	.0	.3	.0	.3	.0	.1	.0	.0	.0	.0	.4	2.7	9.8
S	.1	.7	.7	4.7	.1	.9	.1	.7	.0	.2	.0	.1	.0	.0	1.1	7.3	9.8
SSW	.1	.4	.6	4.2	.2	1.0	.1	.7	.0	.1	.0	.0	.0	.0	1.0	6.5	10.0
SW	.1	.9	.7	5.1	.2	1.2	.1	.9	.0	.2	.0	.0	.0	.0	1.2	8.3	9.9
WSW	.1	.4	.4	2.4	.1	.7	.1	.5	.0	.2	.0	.0	.0	.0	.6	4.2	10.3
W	.1	.6	.3	2.0	.1	.5	.1	.4	.0	.2	.0	.0	.0	.0	.5	3.6	9.6
WNW	.0	.2	.2	1.4	.1	.4	.0	.3	.0	.2	.0	.0	.0	.0	.4	2.7	11.4
NW	.1	.4	.3	1.9	.1	.7	.1	.7	.0	.3	.0	.1	.0	.0	.6	4.1	11.8
NNW	.0	.1	.3	1.8	.1	.8	.1	.9	.1	.5	.0	.1	.0	.1	.6	4.2	13.5
CALM	.2	1.4													.2	1.4	
TOTAL	1.2	8.1	7.1	48.5	2.4	16.4	2.3	15.9	1.2	8.0	.4	2.6	.1	.6	14.7	100	11.9

ORF NORFOLK, VA

CLASS 2 /NIGHT

CEILING-VISIBILITY

WIND GRAPH



# CEILING-VISIBILITY WIND TABULATIONS

ANNUAL

STATION NAME: ORF NORFOLK, VA

PERIOD: 1948-1978

CLASS 2 / NIGHT

STATION NUMBER: 13737

NO. OBS.: 5543

D I R	SPEED GROUPS (MPH)														AVG WIND SPEED		
	0-3		4-12		13-15		16-18		19-24		25-31		32+			TOTAL	
	A	B	A	B	A	B	A	B	A	B	A	B	A	B		C	D
N	.1	.9	.6	4.5	.3	2.2	.3	2.0	.2	1.7	.1	.6	.0	.2	1.6	12.1	13.8
NNE	.1	.4	.4	3.2	.2	1.7	.3	2.2	.2	1.7	.1	.6	.0	.1	1.4	10.0	15.0
NE	.1	.9	.7	5.2	.3	2.3	.4	2.7	.2	1.4	.1	.4	.0	.1	1.8	13.1	13.1
ENE	.1	.5	.5	3.9	.2	1.1	.1	.9	.1	.5	.0	.3	.0	.1	1.0	7.4	11.9
E	.1	.8	.4	2.9	.1	.4	.1	.5	.0	.2	.0	.1	.0	.0	.7	4.9	9.5
ESE	.1	.6	.2	1.6	.0	.3	.0	.2	.0	.2	.0	.0	.0	.0	.4	2.9	8.8
SE	.1	.9	.3	2.1	.0	.3	.0	.3	.0	.1	.0	.0	.0	.0	.5	3.8	8.5
SSE	.1	.6	.3	1.9	.0	.2	.0	.3	.0	.1	.0	.0	.0	.0	.4	3.2	8.6
S	.2	1.3	.5	3.8	.1	.6	.1	.8	.0	.3	.0	.0	.0	.0	.9	6.8	9.0
SSW	.1	.8	.5	3.3	.1	.6	.1	.4	.0	.2	.0	.0	.0	.0	.7	5.4	9.2
SW	.1	1.1	.5	4.0	.1	1.0	.1	.5	.0	.1	.0	.1	.0	.0	.9	6.7	8.9
WSW	.1	.5	.3	2.1	.1	.6	.0	.3	.0	.1	.0	.1	.0	.0	.5	3.6	9.8
W	.1	.8	.3	2.2	.1	.5	.1	.4	.0	.2	.0	.0	.0	.0	.6	4.1	9.6
WNW	.0	.4	.2	1.6	.1	.4	.1	.5	.0	.3	.0	.1	.0	.0	.4	3.3	11.4
NW	.1	.6	.3	2.1	.1	.7	.1	.8	.1	.5	.0	.1	.0	.0	.7	4.9	11.5
NNW	.1	.4	.3	1.9	.1	.9	.1	1.0	.1	.8	.0	.2	.0	.0	.7	5.1	13.4
CALM	.4	2.8												.4	2.8		
TOTAL	1.9	14.2	6.4	46.5	1.9	14.0	1.9	13.7	1.2	8.4	.4	2.6	.1	.6	13.7	100	11.1



# CEILING-VISIBILITY WIND TABULATIONS

ANNUAL

STATION NAME: ORF NORFOLK, VA

PERIOD: 1948-1978

CLASS 3 / DAY

STATION NUMBER: 13737

NO. OBS.: 5076

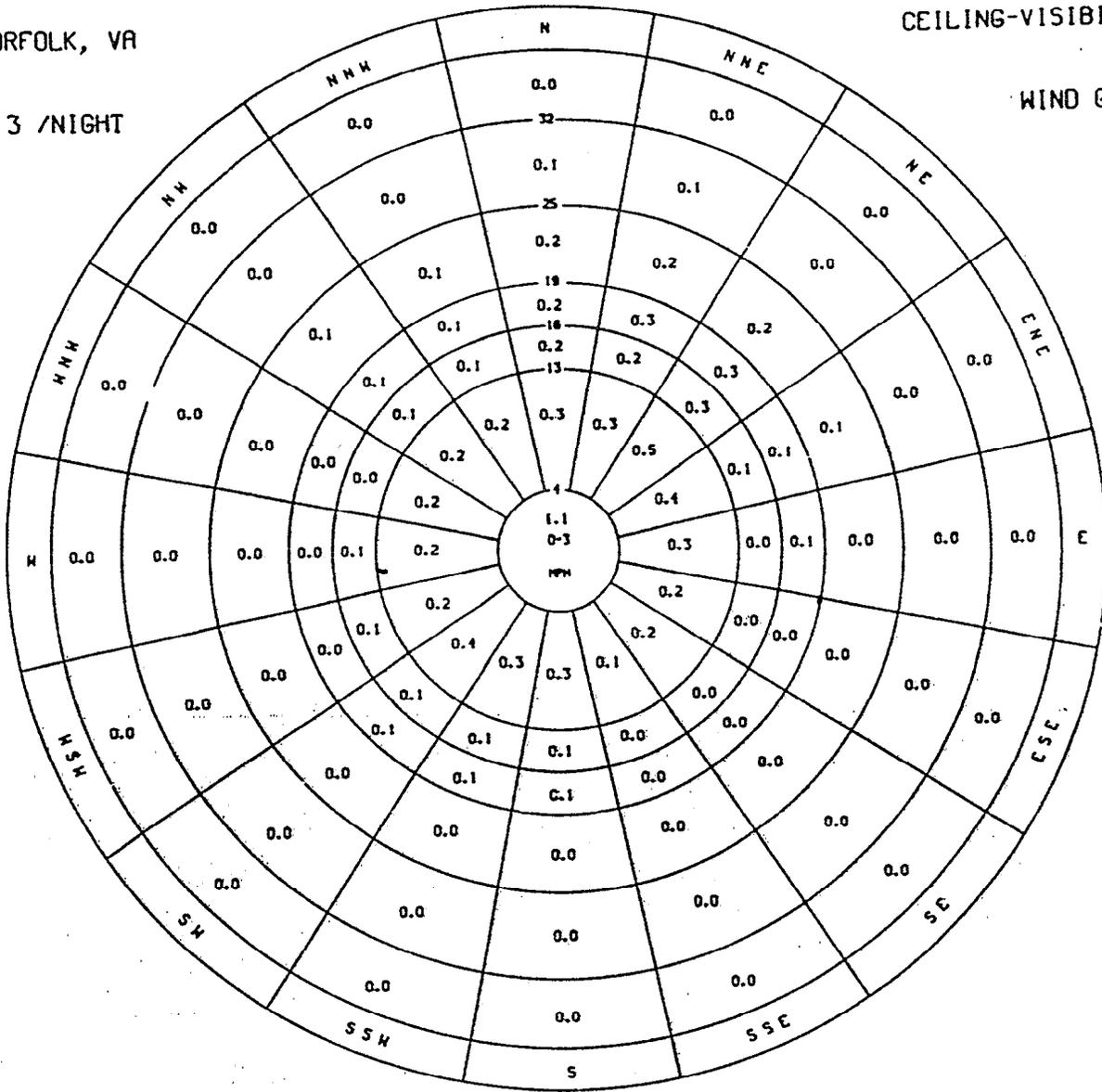
D I R	SPEED GROUPS (MPH)												AVG WIND SPEED				
	0-3		4-12		13-15		16-18		19-24		25-31			32+		TOTAL	
	A	B	A	B	A	B	A	B	A	B	A	B		A	B	C	D
N	0	3	5	4.4	3	2.1	3	2.7	2	1.8	1	6	0	0	1.4	11.9	14.3
NNE	0	3	4	3.6	2	1.8	3	2.3	2	1.3	1	7	0	0	1.2	10.0	14.6
NE	0	3	6	5.2	3	2.8	4	3.2	2	1.5	1	6	0	1	1.7	13.8	14.3
ENE	0	2	4	3.4	2	2.0	2	1.8	1	8	0	2	0	2	1.0	8.6	13.9
E	0	4	3	2.7	1	6	1	6	0	3	0	0	0	0	6	4.6	11.0
ESE	0	2	2	1.4	1	4	0	4	0	2	0	0	0	0	3	2.7	10.9
SE	0	3	2	1.7	0	3	0	3	0	3	0	0	0	0	4	3.1	10.4
SSE	0	3	2	1.6	0	3	0	3	0	1	0	0	0	0	3	2.6	10.4
S	1	6	6	4.7	1	1.0	1	8	0	3	0	1	0	0	9	7.5	10.3
SSW	0	3	5	4.0	1	1.2	1	8	0	2	0	0	0	0	8	6.6	10.5
SW	1	8	6	5.0	2	1.3	1	1.1	0	2	0	0	0	0	1.0	8.5	10.2
WSW	0	3	3	2.4	1	8	1	6	0	2	0	1	0	0	5	4.4	10.7
W	1	5	2	1.9	1	5	0	4	0	2	0	0	0	0	4	3.5	9.9
WNW	0	2	2	1.3	1	5	0	3	0	3	0	0	0	0	3	2.7	11.8
NW	0	3	2	1.9	1	8	1	7	0	4	0	1	0	0	5	4.2	12.2
NNW	0	1	2	1.6	1	9	1	9	1	6	0	1	0	1	5	4.1	13.8
CALM	1	1.1													1	1.1	
TOTAL	8	6.8	5.7	46.8	2.1	17.3	2.1	17.3	1.0	8.6	3	2.7	1	5	12.1	100	12.3

ORF NORFOLK, VA

CLASS 3 /NIGHT

CEILING-VISIBILITY

WIND GRAPH



# CEILING-VISIBILITY WIND TABULATIONS

ANNUAL

STATION NAME: ORF NORFOLK, VA

PERIOD: 1948-1978

CLASS 3 / NIGHT

STATION NUMBER: 13737

NO. OBS.: 4039

D I R	SPEED GROUPS (MPH)														AVG WIND SPEED		
	0-3		4-12		13-15		16-18		19-24		25-31		32+			TOTAL	
	A	B	A	B	A	B	A	B	A	B	A	B	A	B		C	D
N	.1	.7	.3	3.2	.2	2.1	.2	2.2	.2	2.0	.1	.7	.0	.1	1.1	11.0	15.1
NNE	.0	.3	.3	2.8	.2	2.0	.3	2.7	.2	2.1	.1	.8	.0	.1	1.1	10.9	16.0
NE	.1	.7	.5	5.1	.3	2.7	.3	3.2	.2	1.9	.0	.5	.0	.1	1.4	14.2	13.9
ENE	.0	.4	.4	4.0	.1	1.3	.1	1.1	.1	.6	.0	.3	.0	.1	.8	7.9	12.5
E	.1	.7	.3	3.1	.0	.5	.1	.6	.0	.3	.0	.1	.0	.0	.5	5.2	10.0
ESE	.1	.5	.2	1.6	.0	.3	.0	.2	.0	.2	.0	.0	.0	.0	.3	2.9	9.5
SE	.1	.8	.2	1.6	.0	.4	.0	.3	.0	.1	.0	.0	.0	.0	.3	3.3	9.3
SSE	.0	.3	.1	1.4	.0	.2	.0	.4	.0	.1	.0	.0	.0	.0	.2	2.5	10.3
S	.1	.7	.3	3.4	.1	.6	.1	1.1	.0	.3	.0	.0	.0	.0	.6	6.2	10.4
SSW	.1	.7	.3	3.2	.1	.8	.1	.5	.0	.3	.0	.0	.0	.0	.5	5.5	10.1
SW	.1	.9	.4	4.2	.1	1.2	.1	.6	.0	.2	.0	.1	.0	.0	.7	7.1	9.8
WSW	.0	.5	.2	1.9	.1	.7	.0	.4	.0	.1	.0	.1	.0	.0	.4	3.6	10.4
W	.1	.5	.2	2.2	.1	.6	.0	.4	.0	.3	.0	.0	.0	.0	.4	4.0	10.3
WNW	.0	.3	.2	1.5	.0	.4	.0	.4	.0	.4	.0	.1	.0	.0	.3	3.2	12.3
NW	.1	.6	.2	2.1	.1	.7	.1	.9	.1	.7	.0	.1	.0	.0	.5	5.1	12.4
NNW	.0	.3	.2	1.6	.1	1.0	.1	1.2	.1	.9	.0	.2	.0	.0	.5	5.3	14.2
CALM	.2	2.0													.2	2.0	
TOTAL	1.1	10.9	4.3	42.8	1.6	15.6	1.6	16.4	1.0	10.4	.3	3.3	.1	.6	10.0	100	12.2



# CEILING-VISIBILITY WIND TABULATIONS

ANNUAL

STATION NAME: ORF NORFOLK, VA

PERIOD: 1948-1978

CLASS 4 / DAY

STATION NUMBER: 13737

NO. OBS.: 856

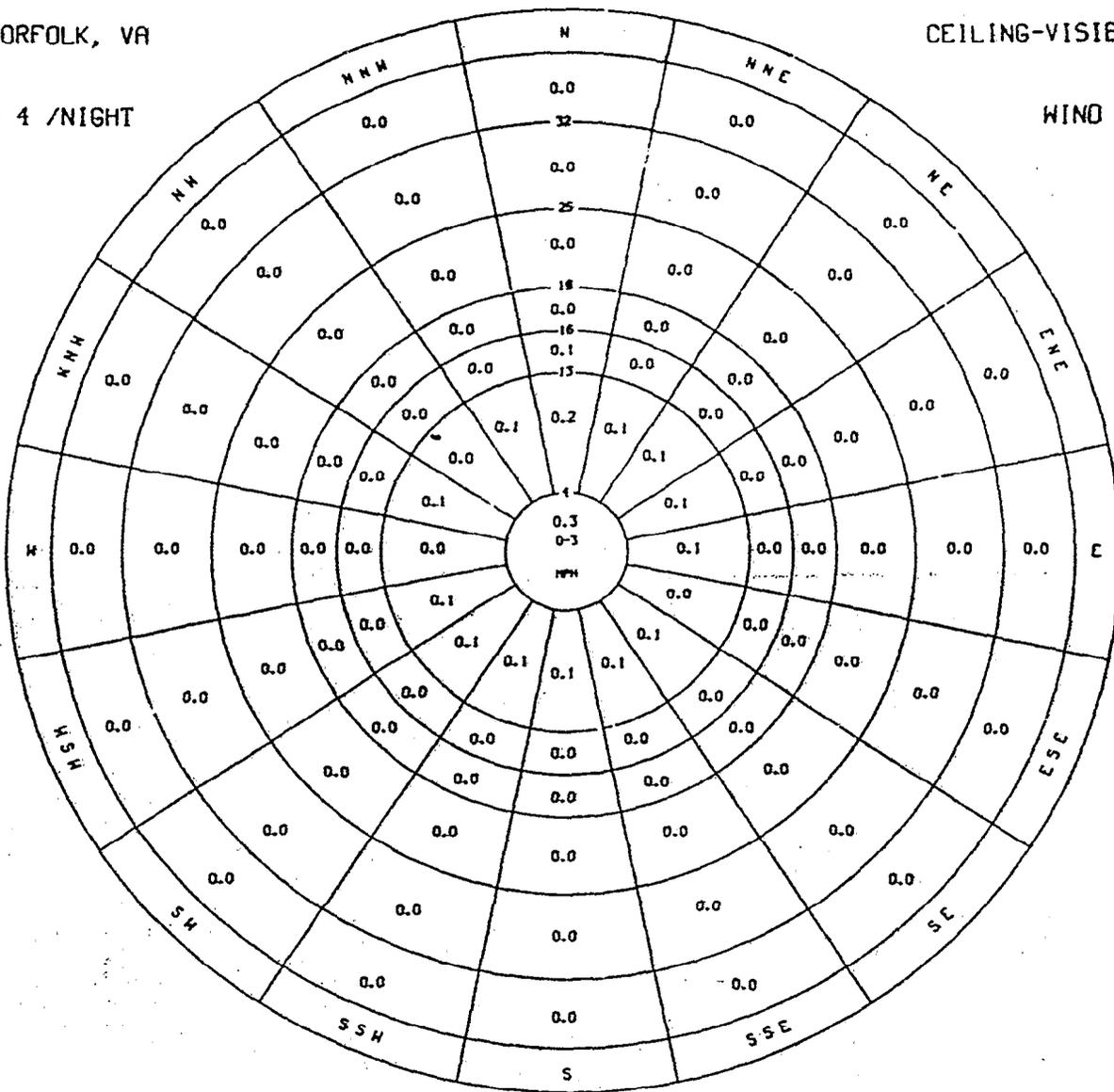
D I R	SPEED GROUPS (MPH)														AVG WIND SPEED		
	0-5		4-12		13-15		16-18		19-24		25-31		32+			TOTAL	
	A	B	A	B	A	B	A	B	A	B	A	B	A	B		C	D
N	.0	.8	.1	8.6	.1	4.0	.0	2.7	.0	2.7	.0	.4	.0	.1	.3	19.3	13.2
NNE	.0	.6	.1	6.3	.0	2.0	.0	2.7	.0	1.4	.0	.4	.0	.0	.2	13.3	12.7
NE	.0	.2	.1	4.4	.0	2.1	.0	2.2	.0	.7	.0	.8	.0	.4	.2	10.9	14.2
ENE	.0	.5	.1	4.2	.0	1.1	.0	1.2	.0	.2	.0	.2	.0	.0	.1	7.4	11.4
E	.0	.5	.0	2.1	.0	.6	.0	.2	.0	.0	.0	.0	.0	.0	.1	3.4	8.7
ESE	.0	.2	.0	1.8	.0	.1	.0	.1	.0	.0	.0	.0	.0	.0	.0	2.2	8.2
SE	.0	1.1	.0	1.4	.0	.2	.0	.0	.0	.0	.0	.0	.0	.0	.0	2.9	8.2
SSE	.0	.5	.0	1.9	.0	.4	.0	.2	.0	.0	.0	.0	.0	.0	.1	4.9	8.0
S	.0	.7	.1	4.0	.0	.1	.0	.1	.0	.0	.0	.0	.0	.0	.1	4.9	8.1
SSH	.0	.5	.1	3.9	.0	.2	.0	.4	.0	.0	.0	.0	.0	.0	.1	6.9	8.7
SW	.0	1.1	.1	4.9	.0	.5	.0	.4	.0	.1	.0	.0	.0	.0	.1	3.4	7.9
WSW	.0	.6	.0	2.3	.0	.4	.0	.1	.0	.0	.0	.0	.0	.0	.1	4.1	8.5
W	.0	.9	.0	2.5	.0	.5	.0	.0	.0	.1	.0	.1	.0	.0	.1	4.1	10.3
WNW	.0	.1	.0	1.9	.0	.4	.0	.2	.0	.1	.0	.1	.0	.0	.0	2.8	11.3
NW	.0	.5	.0	2.6	.0	.2	.0	.5	.0	.0	.0	.1	.0	.2	.1	4.1	13.5
NNW	.0	.1	.0	2.7	.0	.7	.0	.8	.0	.6	.0	.2	.0	.0	.1	5.1	13.5
CALM	.0	1.8													.0	1.8	
TOTAL	.2	10.5	.9	55.4	.2	13.3	.2	11.8	.1	6.0	.0	2.3	.0	.7	1.7	100	10.9

ORF NORFOLK, VA

CLASS 4 /NIGHT

CEILING-VISIBILITY

WIND GRAPH



# CEILING-VISIBILITY WIND TABULATIONS

ANNUAL

STATION NAME: ORF NORFOLK, VA

PERIOD: 1948-1978

CLASS 4 / NIGHT

STATION NUMBER: 13737

NO. OBS.: 838

D I R	SPEED GROUPS (MPH)														AVG WIND SPEED				
	0-3		4-12		13-15		16-18		19-24		25-31		32+			TOTAL			
	A	B	A	B	A	B	A	B	A	B	A	B	A	B		C	D		
N	0	1.0	2	9.4	1	2.9	0	2.1	0	1.8	0	2	0	1	4	17.5	11.9		
NNE	0	5	1	3.8	0	1.1	0	1.1	0	8	0	2	0	0	2	7.5	12.0		
NE	0	5	1	6.0	0	1.6	0	1.8	0	6	0	2	0	0	2	10.6	11.3		
ENE	0	6	1	4.2	0	7	0	6	0	5	0	2	0	2	1	7.0	11.5		
E	0	1.1	1	3.0	0	4	0	5	0	1	0	1	0	0	1	5.1	8.4		
ESE	0	4	0	1.8	0	4	0	2	0	1	0	0	0	0	1	2.9	9.0		
SE	0	4	1	3.2	0	0	0	1	0	1	0	0	0	0	1	3.8	8.4		
SSE	0	1.0	1	2.7	0	2	0	1	0	0	0	0	0	0	1	4.1	7.4		
S	0	1.4	1	4.3	0	7	0	1	0	0	0	0	0	0	1	6.6	7.6		
SSH	0	1.0	1	3.0	0	4	0	0	0	1	0	0	0	0	1	4.4	7.1		
SW	0	1.2	1	3.0	0	5	0	2	0	0	0	0	0	0	1	4.9	6.9		
WSW	0	6	1	2.7	0	6	0	2	0	0	0	0	0	0	1	4.2	8.6		
W	0	8	0	2.4	0	6	0	2	0	2	0	0	0	0	1	4.3	9.3		
WNW	0	6	1	2.5	0	6	0	8	0	0	0	0	0	0	1	4.5	9.8		
NW	0	5	0	2.4	0	1.2	0	5	0	0	0	0	0	0	1	4.5	9.8		
NNW	0	2	1	3.5	0	8	0	4	0	6	0	2	0	0	1	5.7	12.2		
CALM	0	2.3													0	2.3			
TOTAL	3	13.8	1	2	57.9	3	12.5	2	9.1	1	5.0	0	1	3	0	4	2	100	9.8



# CEILING-VISIBILITY WIND TABULATIONS

ANNUAL

STATION NAME: ORF NORFOLK, VA

PERIOD: 1948-1978

CLASS 5 / DAY

STATION NUMBER: 13737

NO. OBS.: 216

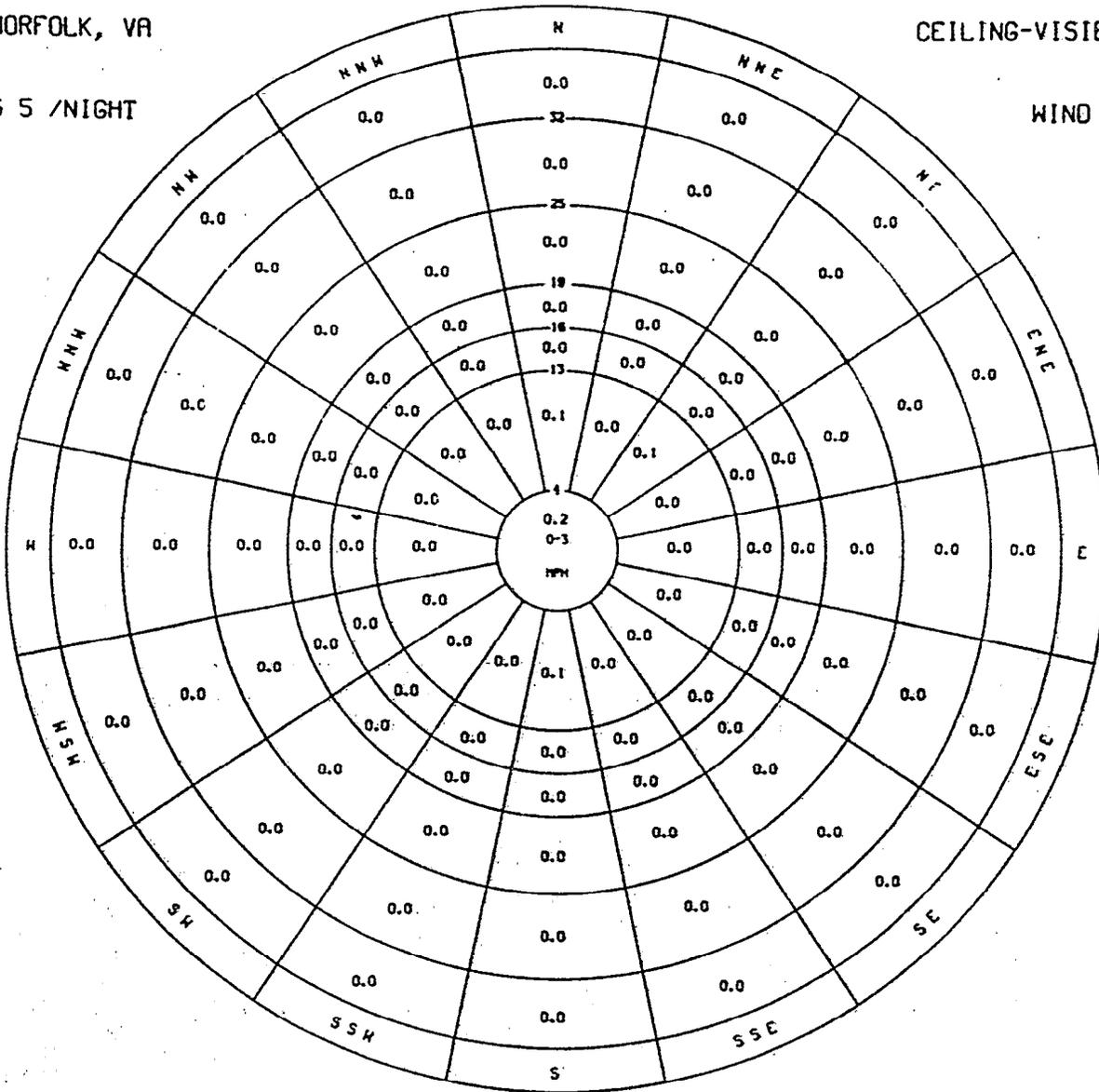
D I R	SPEED GROUPS (MPH)												AVG WIND SPEED				
	0-3		4-12		13-15		16-18		19-24		25-31			32+		TOTAL	
	A	B	A	B	A	B	A	B	A	B	A	B		A	B	C	D
N	0	0	1	13.0	0	4.2	0	2.3	0	1.4	0	5	0	5	1	21.8	12.3
NNE	0	1.9	0	7.9	0	1.9	0	5	0	1.4	0	0	0	0	1	13.4	9.7
NE	0	1.9	0	7.4	0	1.9	0	1.9	0	1.9	0	0	0	0	1	14.8	10.9
ENE	0	5	0	4.2	0	5	0	0	0	9	0	0	0	0	0	6.0	10.8
E	0	0	0	9	0	0	0	0	0	0	0	0	0	0	0	9	7.5
ESE	0	9	0	1.4	0	0	0	0	0	0	0	0	0	0	0	2.3	5.0
SE	0	9	0	0	0	5	0	0	0	0	0	0	0	0	0	1.4	7.0
SSE	0	9	0	1.9	0	0	0	0	0	0	0	0	0	0	0	2.8	6.5
S	0	5	0	4.6	0	1.4	0	0	0	0	0	0	0	0	0	6.5	7.9
SSW	0	1.4	0	3.7	0	0	0	0	0	0	0	0	0	0	0	5.1	6.4
SW	0	9	0	5.6	0	0	0	0	0	0	0	0	0	0	0	6.5	7.6
WSW	0	9	0	1.4	0	5	0	0	0	0	0	0	0	0	0	2.8	6.5
W	0	5	0	2.3	0	5	0	5	0	5	0	0	0	0	0	4.2	10.9
WNW	0	5	0	1.9	0	5	0	0	0	0	0	0	0	0	0	2.8	8.3
NW	0	5	0	1.4	0	0	0	9	0	0	0	0	0	0	0	2.8	9.8
NNW	0	0	0	3.2	0	5	0	0	0	0	0	0	0	0	0	3.7	9.5
CALM	0	2.3													0	2.3	
TOTAL	1	14.4	3	60.6	1	12.0	0	6.0	0	6.0	0	5	0	5	4	100	9.5

ORF NORFOLK, VA

CLASS 5 /NIGHT

CEILING-VISIBILITY

WIND GRAPH



# CEILING-VISIBILITY WIND TABULATIONS

ANNUAL

STATION NAME: ORF NORFOLK, VA

PERIOD: 1949-1978

CLASS 5 / NIGHT

STATION NUMBER: 13737

NO. OBS.: 342

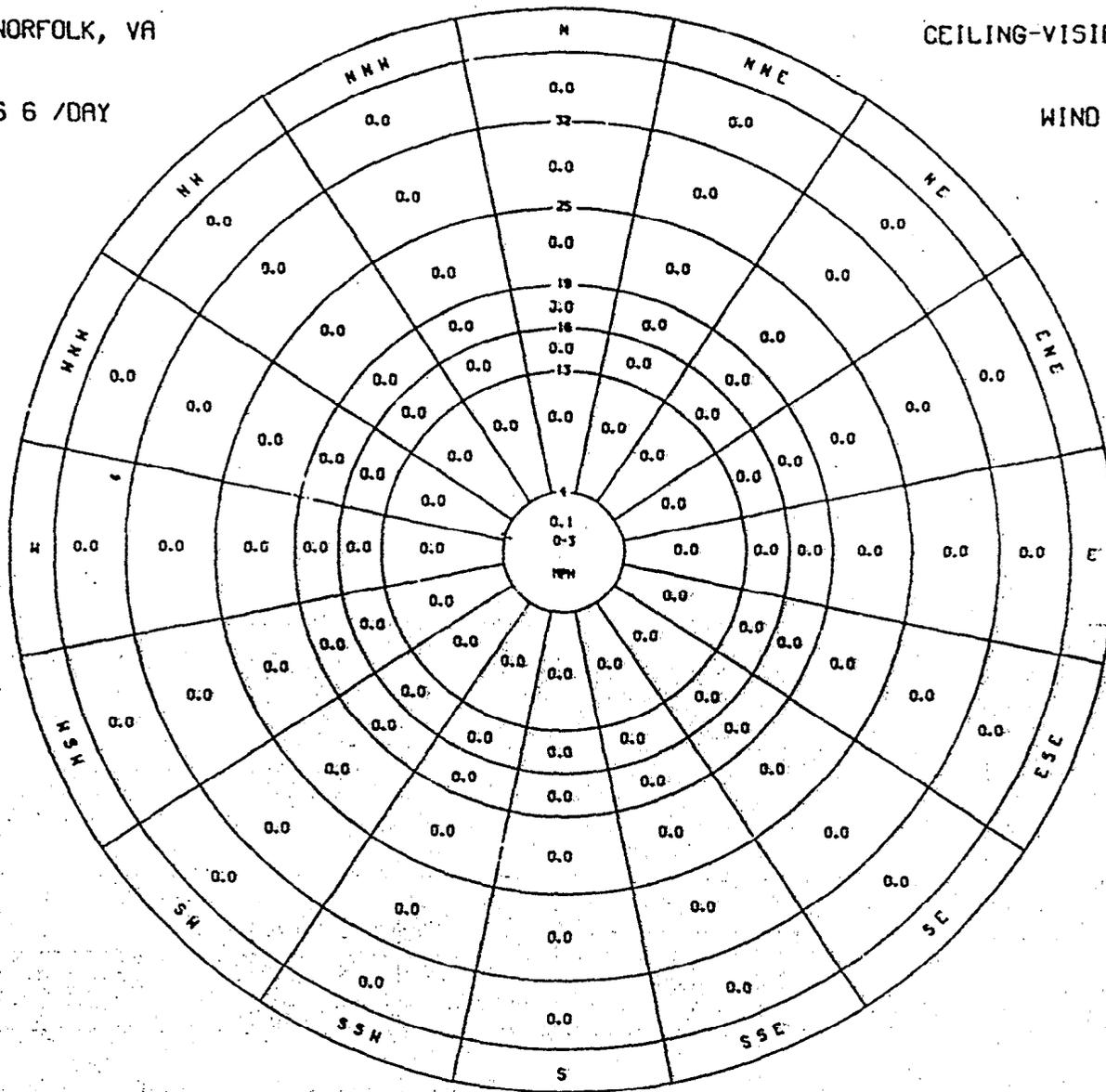
D I R	SPEED GROUPS (MPH)																AVG WIND SPEED
	0-3		4-12		13-15		16-18		19-24		25-31		32+		TOTAL		
	A	B	A	B	A	B	A	B	A	B	A	B	A	B	C	D	
N	0	1.5	1	8.5	0	3.2	0	.9	0	.3	0	.6	0	.3	1	15.2	11.1
NNE	0	.3	0	5.6	0	.6	0	1.2	0	.3	0	.3	0	.3	1	8.5	11.7
NE	0	1.2	1	6.1	0	1.8	0	1.2	0	.0	0	.0	0	.0	1	10.2	9.2
ENE	0	.6	0	3.5	0	.3	0	.3	0	.0	0	.0	0	.0	0	4.7	8.1
E	0	1.5	0	1.5	0	.0	0	.3	0	.0	0	.0	0	.0	0	3.2	5.7
ESE	0	1.2	0	1.2	0	.0	0	.0	0	.0	0	.0	0	.0	0	2.3	4.2
SE	0	1.8	0	3.5	0	.9	0	.0	0	.0	0	.0	0	.0	1	6.1	6.4
SSE	0	2.0	0	3.5	0	.3	0	.0	0	.0	0	.0	0	.0	0	5.8	5.9
S	0	2.3	1	7.6	0	.6	0	.0	0	.0	0	.0	0	.0	1	10.5	6.7
SSW	0	.9	0	4.4	0	.3	0	.0	0	.0	0	.0	0	.0	0	5.6	6.4
SW	0	1.2	0	3.2	0	.3	0	.0	0	.0	0	.0	0	.0	0	4.7	6.4
WSW	0	.0	0	2.0	0	.3	0	.0	0	.0	0	.0	0	.0	0	2.3	9.1
W	0	2.3	0	3.5	0	.3	0	.0	0	.0	0	.0	0	.0	1	6.1	6.3
WNW	0	.9	0	1.8	0	.0	0	.0	0	.3	0	.0	0	.0	0	2.9	8.0
NW	0	.3	0	2.6	0	.6	0	.3	0	.0	0	.0	0	.0	0	3.8	8.2
NNW	0	.3	0	1.2	0	.3	0	.3	0	.3	0	.0	0	.0	0	2.3	11.1
CALM	0	5.6													0	5.6	
TOTAL	2	23.7	5	59.6	1	9.6	0	4.4	0	1.2	0	.9	0	.6	8	100	7.8

ORF NORFOLK, VA

CLASS 6 /DAY

CEILING-VISIBILITY

WIND GRAPH



# CEILING-VISIBILITY WIND TABULATIONS

ANNUAL

STATION NAME: ORF NORFOLK, VA

PERIOD: 1948-1978

CLASS 6 / DAY

STATION NUMBER: 13737

NO. OBS.: 196

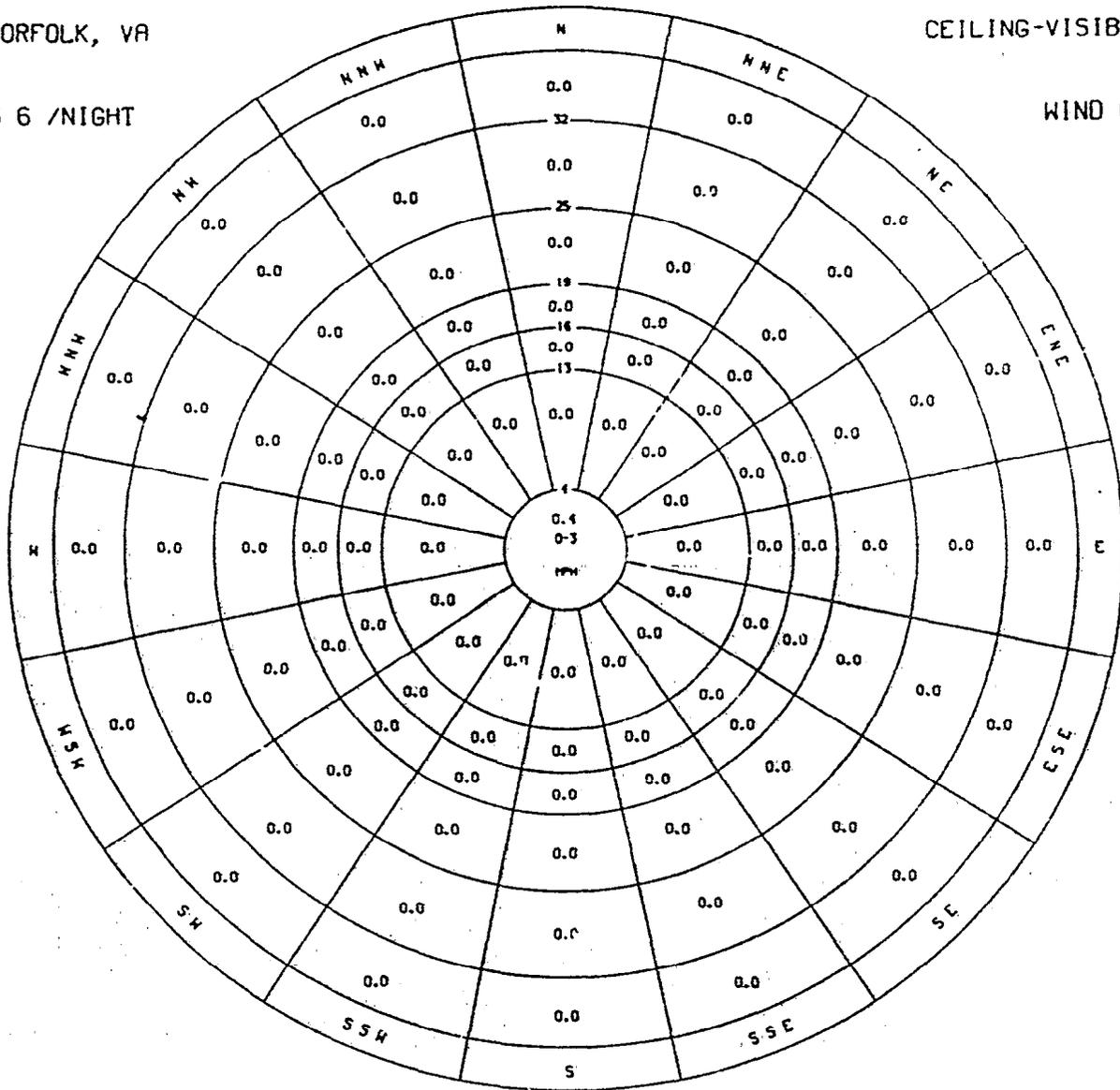
D I R	SPEED GROUPS (MPH)														AVG WIND SPEED		
	0-3		4-12		13-15		16-18		19-24		25-31		32+			TOTAL	
	A	B	A	B	A	B	A	B	A	B	A	B	A	B		C	D
N	.0	.5	.0	10.7	.0	1.5	.0	1.5	.0	.0	.0	1.0	.0	.0	.1	15.3	10.3
NNE	.0	.0	.0	5.1	.0	1.0	.0	.0	.0	.5	.0	.5	.0	.0	.0	7.1	11.6
NE	.0	.5	.0	3.1	.0	1.0	.0	.0	.0	.0	.0	.0	.0	.0	.0	4.6	8.2
ENE	.0	2.6	.0	4.6	.0	.0	.0	.0	.0	.0	.0	.0	.0	.0	.0	7.1	5.8
E	.0	1.5	.0	1.5	.0	.0	.0	.0	.0	.0	.0	.0	.0	.0	.0	3.1	5.2
ESE	.0	.5	.0	1.0	.0	.0	.0	.0	.0	.0	.0	.0	.0	.0	.0	1.5	5.0
SE	.0	1.0	.0	.0	.0	.0	.0	.0	.0	.0	.0	.0	.0	.0	.0	1.0	2.5
SSE	.0	1.5	.0	2.0	.0	.0	.0	.0	.0	.0	.0	.0	.0	.0	.0	3.6	4.7
S	.0	4.1	.0	7.1	.0	.0	.0	.0	.0	.0	.0	.0	.0	.0	.0	11.2	5.0
SSH	.0	2.0	.0	10.2	.0	.0	.0	.0	.0	.0	.0	.0	.0	.0	.0	12.2	5.9
SW	.0	3.1	.0	6.1	.0	.0	.0	.0	.0	.0	.0	.0	.0	.5	.0	9.7	6.7
WSW	.0	1.0	.0	3.6	.0	.0	.0	.0	.0	.0	.0	.0	.0	.0	.0	4.6	6.9
W	.0	2.6	.0	1.5	.0	.0	.0	.0	.0	.0	.0	.0	.0	.0	.0	4.1	3.5
WNW	.0	.5	.0	.5	.0	.0	.0	.0	.0	.0	.0	.0	.0	.0	.0	1.0	4.0
NW	.0	2.6	.0	.5	.0	.0	.0	.0	.0	.0	.0	.0	.0	.0	.0	3.1	3.2
NNW	.0	.5	.0	2.6	.0	1.0	.0	.5	.0	.0	.0	.0	.0	.0	.0	4.6	9.0
CALM	.0	6.1													.0	6.1	
TOTAL	.1	30.6	.2	60.2	.0	4.6	.0	2.0	.0	.5	.0	1.5	.0	.5	.4	100	6.6

ORF NORFOLK, VA

CLASS 6 /NIGHT

CEILING-VISIBILITY

WIND GRAPH



# CEILING-VISIBILITY WIND TABULATIONS

ANNUAL

STATION NAME: ORF NORFOLK, VA

PERIOD: 1948-1978

CLASS 6 / NIGHT

STATION NUMBER: 13737

NO. OBS.: 324

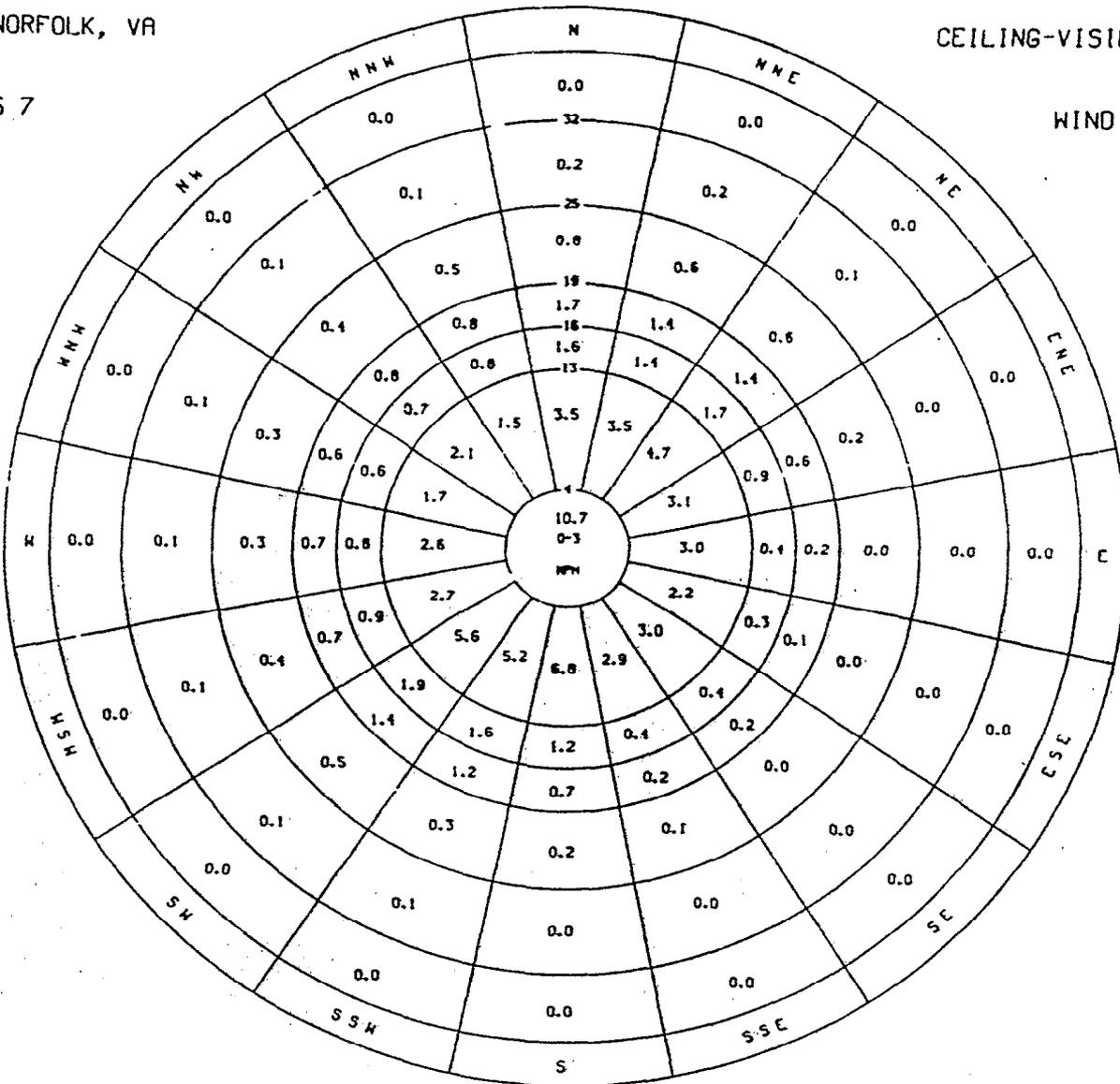
D I R	SPEED GROUPS (MPH)														AVG WIND SPEED		
	0-3		4-12		13-15		16-18		19-24		25-31		32+			TOTAL	
	A	B	A	B	A	B	A	B	A	B	A	B	A	B		C	D
N	0	2.5	0	3.4	0	3	0	6	0	0	0	0	0	3	1	7.1	7.9
NNE	0	1.2	0	4.6	0	6	0	0	0	0	0	0	0	0	1	6.5	7.1
NE	0	3.4	0	4.3	0	3	0	3	0	0	0	0	0	0	1	8.3	6.5
ENE	0	1.5	0	3.4	0	9	0	0	0	0	0	0	0	0	0	5.9	6.4
E	0	9	0	1.5	0	0	0	0	0	0	0	0	0	0	0	2.5	4.9
ESE	0	2.2	0	1.2	0	0	0	3	0	0	0	0	0	0	0	3.7	5.4
SE	0	2.5	0	4.6	0	0	0	0	0	0	0	0	0	0	1	7.1	5.3
SSE	0	2.2	0	4.9	0	0	0	0	0	0	0	0	0	0	1	7.1	5.9
S	1	7.1	0	3.7	0	6	0	0	0	0	0	0	0	0	1	11.4	4.4
SSW	0	2.2	0	4.6	0	0	0	0	0	0	0	0	0	0	1	6.8	5.5
SW	0	3.1	0	4.9	0	0	0	0	0	0	0	0	0	0	1	8.0	5.0
WSW	0	3	0	3.1	0	0	0	0	0	0	0	0	0	0	0	3.4	5.5
W	0	1.5	0	1.2	0	0	0	3	0	0	0	0	0	0	0	3.1	5.7
WNW	0	0	0	9	0	0	0	0	0	0	0	0	0	0	0	9	7.3
NW	0	2.5	0	1.2	0	0	0	0	0	0	0	0	0	0	0	3.7	4.3
NNW	0	1.5	0	1.2	0	0	0	0	0	0	0	0	0	0	0	2.8	4.3
CALM	1	11.7													1	11.7	
TOTAL	4	46.3	4	49.1	0	2.8	0	1.5	0	0	0	0	0	3	8	100	5.0

ORF NORFOLK, VA

CLASS 7

CEILING-VISIBILITY

WIND GRAPH



FEDERAL CLIMATE COMPLEX ASHEVILLE

INTERNATIONAL STATION METEOROLOGICAL CLIMATE SUMMARY

STA 723085 ; KNGU ; NORFOLK NAS ,VA,US

LAT 36 56N :LONG 076 17W :ELEV 16(ft) 5(m) :TYPE NAVY SMOS V2.1 02071992

- Cumulative Percent Frequency Daily MAXIMUM TEMPERATURES

OR: 1945-1990

emp(F)	JAN	FEB	MAR	APR	MAY	JUN	JUL	AUG	SEP	OCT	NOV	DEC	ANN
>=100	0	0	0	0	0	.3	.2	.5	.1	0	0	0	.1
>=95	0	0	0	0	.4	5.6	10.3	7.2	1.2	0	0	0	2.1
>=90	0	0	.1	1.0	5.4	22.8	36.9	27.8	9.3	.4	0	0	8.7
>=85	0	0	1.1	5.9	17.7	45.7	64.4	56.0	25.9	3.9	0	0	18.5
>=80	0	.4	4.1	14.3	35.3	69.1	88.2	80.9	50.4	12.1	2.2	0	29.9
>=75	1.0	2.1	9.8	27.5	55.1	86.0	98.6	97.2	77.3	28.9	9.9	1.1	41.4
>=70	3.9	6.4	18.1	41.2	72.9	96.7	99.9	99.9	93.4	51.1	22.1	5.8	51.1
>=65	10.7	13.2	29.0	56.8	88.1	99.3	100	100	98.7	73.9	36.8	14.7	60.3
>=60	21.1	23.6	41.8	75.6	96.7	99.9	100	100	99.9	92.1	55.2	26.0	69.5
>=55	30.3	35.5	55.9	89.6	99.6	100	100	100	100	98.0	72.6	36.7	76.7
>=50	41.4	47.7	72.3	97.5	100	100	100	100	100	99.3	88.4	54.3	83.5
>=45	57.7	62.8	87.6	99.6	100	100	100	100	100	100	95.8	72.8	89.8
>=40	75.8	81.8	96.3	100	100	100	100	100	100	100	99.2	86.3	95.0
>=35	88.6	93.1	98.9	100	100	100	100	100	100	100	99.9	94.8	98.0
>=30	96.8	98.7	99.8	100	100	100	100	100	100	100	100	98.9	99.5
>=25	99.4	99.8	100	100	100	100	100	100	100	100	100	99.7	99.9
>=20	99.8	100	100	100	100	100	100	100	100	100	100	99.9	100
>=15	99.9	100	100	100	100	100	100	100	100	100	100	100	100
>=10	100	100	100	100	100	100	100	100	100	100	100	100	100
MEAN	48.3	50.3	57.7	67.5	75.5	83.2	86.9	85.5	79.8	69.9	61.0	51.6	68.2
STDV	11.6	11.6	11.7	10.3	8.8	7.5	5.9	6.0	6.9	7.9	9.7	10.9	16.4
#OBS	1395	1287	1423	1380	1426	1380	1393	1426	1350	1394	1378	1426	16658

PERCENT < .05  
EXCESSIVE MISSING DATA - VALUE NOT COMPUTED

FEDERAL CLIMATE COMPLEX ASHEVILLE  
-----INTERNATIONAL STATION METEOROLOGICAL CLIMATE SUMMARY-----

:STA 723085 ; KNGU ; NORFOLK NAS ,VA,US  
LAT 36 56N :LONG 076 17W :ELEV 16(ft) 5(m) :TYPE NAVY SHOS V2.1 02071992  
- Cumulative Percent Frequency Daily MEAN TEMPERATURES

OR: : 1945-1990

Temp(F)	JAN	FEB	MAR	APR	MAY	JUN	JUL	AUG	SEP	OCT	NOV	DEC	ANN
>=90	0	0	0	0	0	.4	.8	.4	0	0	0	0	.1
>=85	0	0	0	0	.1	5.8	15.6	10.7	2.0	0	0	0	2.9
>=80	0	0	0	.1	4.6	28.2	55.1	45.8	15.6	.8	0	0	12.6
>=75	0	0	.8	3.4	18.1	58.9	86.7	82.3	44.0	5.3	.2	0	25.1
>=70	0	.4	2.8	11.8	41.1	82.3	99.0	97.8	75.5	19.0	3.0	.1	36.2
>=65	1.1	1.6	8.1	25.3	64.9	96.8	99.9	99.8	93.8	43.1	11.0	1.8	45.7
>=60	3.9	4.8	16.2	44.1	85.8	99.4	100	100	99.4	69.9	24.8	6.0	54.7
>=55	9.2	12.0	28.1	67.0	97.1	100	100	100	99.9	89.3	46.5	16.3	63.9
>=50	21.6	24.3	46.2	88.6	99.4	100	100	100	100	97.6	67.8	28.4	73.0
>=45	34.0	40.2	70.5	97.6	100	100	100	100	100	99.5	86.0	47.5	81.4
>=40	55.5	60.0	87.8	99.6	100	100	100	100	100	100	95.9	70.5	89.3
>=35	74.3	81.3	96.8	100	100	100	100	100	100	100	99.3	86.5	94.9
>=30	89.5	93.5	99.2	100	100	100	100	100	100	100	99.9	95.0	98.1
>=25	97.1	99.1	99.9	100	100	100	100	100	100	100	100	98.9	99.6
>=20	99.4	99.8	100	100	100	100	100	100	100	100	100	99.8	99.9
>=15	99.9	100	100	100	100	100	100	100	100	100	100	99.9	100
>=10	99.9	100	100	100	100	100	100	100	100	100	100	100	100
>=5	100	100	100	100	100	100	100	100	100	100	100	100	100
MEAN	41.3	42.8	49.8	58.8	67.5	75.6	79.8	78.9	73.5	63.3	53.7	44.6	60.9
STDV	9.6	9.3	9.3	8.1	7.1	6.9	4.4	4.6	5.7	7.0	8.3	9.2	15.6
#OBS	1395	1286	1423	1380	1426	1380	1393	1426	1350	1394	1378	1426	16657

\* = PERCENT < .05  
# = EXCESSIVE MISSING DATA - VALUE NOT COMPUTED

-----INTERNATIONAL STATION METEOROLOGICAL CLIMATE SUMMARY-----

:STA 723086 | KMGU | NORFOLK NAS ,VA,US

:LAT 36 56N :LONG 076 17W :ELEV 16(ft) 5(m) :TYPE NAVY SHOS V2.1 02071992

37 - STATION CLIMATIC SUMMARY

POR: (HOURLY): 1945-1990

TEMPERATURE (DEG F)		PRECIPITATION (INCHES) (*)				REL HUM:VAP:DEW: PR				WIND (KTS)				MEAN NO. OF DAYS WITH (&)									
MEANS		EXTREME		PRECIP.		SNOWFALL (#)		PERCENT:PR		PT. ALT		SKY		PRECIP.		SNOW-		TEMP (DEG F)					
MAX	MIN	MAX	MIN	24H	MEAN	24H	(LST)	AM	PM	HTG.	FT.	PREVAIL	MAX	CVR	INCHES	FALL(*)	TH	FOG	MAX	MAX	MIN	MIN	

JAN	48	34	41	78	-1	3.2	8.9	.9	2.6	2	12	7	71	59	.16	29	60	NNE	11	55	OVR	10	2	1	1	#	13	0	#	14	1
FEB	50	35	43	82	12	3.2	6.3	.6	2.7	3	25	13	71	57	.17	30	70	NNE	12	58	OVR	10	2	1	#	1	12	0	1	11	#
MAR	58	41	50	90	20	3.2	9.1	.4	2.4	1	19	11	73	55	.22	36	65	SSW	11	59	OVR	10	2	#	#	2	12	#	3	4	0
APR	67	50	59	93	29	2.8	6.7	.5	2.7	T	T	T	71	52	.29	44	65	SSW	10	61	OVR	10	2	#	0	3	11	#	8	#	0
MAY	76	59	68	97	37	3.3	9.3	.6	2.8	0	0	0	75	57	.43	55	45	SSW	9	59	OVR	10	2	0	0	5	14	2	17	0	0
JUN	83	67	76	101	47	3.3	9.6	.4	5.2	0	0	0	77	58	.59	64	40	SSW	9	61	SCT	9	2	0	0	6	12	7	26	0	0
JUL	87	72	80	100	58	4.5	12.2	1.0	6.5	0	0	0	80	61	.71	69	30	SSW	9	65	SCT	11	3	0	0	8	12	11	31	0	0
AUG	85	72	79	102	52	4.4	11.8	.9	6.4	0	0	0	82	63	.70	58	25	SSW	8	70	SCT	10	3	0	0	7	15	9	30	0	0
SEP	80	67	74	100	50	3.9	18.2	.5	7.9	0	0	0	80	62	.57	63	35	ESE	9	66	SCT	7	2	0	0	3	13	3	23	0	0
OCT	70	56	63	93	33	2.7	9.5	.2	3.3	0	0	0	78	60	.39	52	45	NNE	12	85	OVR	7	2	0	0	1	14	#	9	0	0
NOV	61	46	54	84	21	2.7	6.3	.4	2.8	T	2	1	75	58	.26	42	55	SSW	9	63	OVR	8	2	#	0	1	13	0	3	2	0
DEC	52	37	45	79	9	2.9	6.6	.5	2.6	1	16	9	72	59	.19	33	60	SSW	9	61	OVR	9	2	#	#	#	13	0	#	10	#
ANN	68	53	61	102	-1	40.2	67.8	21.8	7.9	7	45	13	75	58	.35	49	55	SSW	10	85	OVR	111	26	2	1	37	154	32	151	40	1
POR	46	46	46	46	46	46	46	46	46	46	46	46	46	46	46	46	44	46	46	43	44	46	46	46	46	38	38	46	46	46	46

- T = TRACE AMOUNTS ( < .05 < .5 INCHES
- # = MEAN NO. DAYS < .5 DAYS
- \$ = PRESSURE ALTITUDE IN TENS OF FEET (I.E. 50 = 500 FEET)
- B = NAVY STATIONS REPORT HAIL AS SNOWFALL; ALSO NWS FROM JULY,1948 - DEC.,1955
- + = THE PREDOMINANT SKY CONDITION\PRECIP > LISTED AMOUNT AND < NEXT WHOLE INCH
- \* = VISIBILITY IS NOT CONSIDERED
- E = ANN TOTALS MAY NOT EQUAL SUM OF MONTHLY VALUES DUE TO ROUNDING
- " = 24 HR MAX PRECIP AND SNOWFALL ARE DAILY TOTALS (MID-NIGHT TO MID-NIGHT)
- I = EXCESSIVE MISSING DATA - VALUE NOT COMPUTED
- " = INCHES

-----FEDERAL CLIMATE COMPLEX ASHEVILLE-----

## -----INTERNATIONAL STATION METEOROLOGICAL CLIMATE SUMMARY-----

:STA 723085 : KNGU : NORFOLK NAS ,VA,US

:LAT 36 56N :LONG 076 17W :ELEV 16(ft) 5(m) :TYPE NAVY SMOS V2.1 02071992

41 - STATION CLIMATIC SUMMARY (CONTINUED)

POR: (HOURLY): 1945-1990

		MEAN NO. OF DAYS WITH (#)										
		PRECIPITATION					OBSTR TO VISION					
		FRZ	HAIL	SMOK	BLOW	DUST	OBS					
		R/OZ	R/DZ	SNOW	SLT	PRCP	HAZE	SNOW	SAND	VIS		

JAN	13	#	4	0	15	18	#	0	22		
FEB	12	#	3	0	14	17	#	#	20		
MAR	15	#	2	#	15	18	#	#	22		
APR	14	0	#	#	14	19	0	#	21		
MAY	15	0	0	#	15	21	0	#	24		
JUN	13	0	0	#	13	22	0	#	24		
JUL	16	0	0	0	15	24	0	#	24		
AUG	14	0	0	0	14	24	0	#	26		
SEP	11	0	0	0	11	20	0	#	22		
OCT	11	0	0	0	11	20	0	0	22		
NOV	12	0	#	#	12	18	0	0	21		
DEC	11	#	2	0	12	18	#	0	21		
ANN	156	#	11	#	161	239	1	1	269		
POR	36	38	38	38	38	38	38	38	38		

# = ANN TOTALS MAY NOT EQUAL SUM OF MONTHLY VALUES DUE TO ROUNDING

I = EXCESSIVE MISSING DATA - VALUE NOT COMPUTED

# = MEAN NO. DAYS &lt; .5 DAYS

-----FEDERAL CLIMATE COMPLEX ASHEVILLE-----

**APPENDIX B**  
**COMPENDIUM METHOD TO-14**

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# COMPENDIUM METHOD T0-14

THE DETERMINATION OF VOLATILE ORGANIC  
COMPOUNDS (VOCs) IN AMBIENT AIR USING  
SUMMA<sup>®</sup> PASSIVATED CANISTER SAMPLING  
AND GAS CHROMATOGRAPHIC ANALYSIS



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## METHOD T014

### DETERMINATION OF VOLATILE ORGANIC COMPOUNDS (VOCs) IN AMBIENT AIR USING SUMMA® PASSIVATED CANISTER SAMPLING AND GAS CHROMATOGRAPHIC ANALYSIS

#### OUTLINE

1. Scope
2. Applicable Documents
3. Summary of Method
4. Significance
5. Definitions
6. Interferences and Limitations
7. Apparatus
  - 7.1 Sample Collection
    - 7.1.1 Subatmospheric Pressure
    - 7.1.2 Pressurized
  - 7.2 Sample Analysis
    - 7.2.1 GC-MS-SCAN Analytical System
    - 7.2.2 GC-MS-SIM Analytical System
    - 7.2.3 GC-Multidetector Analytical System
  - 7.3 Canister Cleaning System
  - 7.4 Calibration System and Manifold
8. Reagents and Materials
9. Sampling System
  - 9.1 System Description
    - 9.1.1 Subatmospheric Pressure Sampling
    - 9.1.2 Pressurized Sampling
    - 9.1.3 All Samplers
  - 9.2 Sampling Procedure
10. Analytical System
  - 10.1 System Description
    - 10.1.1 GC-MS-SCAN System
    - 10.1.2 GC-MS-SIM System
    - 10.1.3 GC-Multidetector (GC-FID-ECD-PID) System
  - 10.2 GC-MS-SCAN-SIM System Performance Criteria
    - 10.2.1 GC-MS System Operation
    - 10.2.2 Daily GC-MS Tuning
    - 10.2.3 GC-MS Calibration
      - 10.2.3.1 Initial Calibration
      - 10.2.3.2 Routine Calibration
  - 10.3 GC-FID-ECD System Performance Criteria (With Optional PID)
    - 10.3.1 Humid Zero Air Certification
    - 10.3.2 GC Retention Time Windows Determination
    - 10.3.3 GC Calibration
      - 10.3.3.1 Initial Calibration
      - 10.3.3.2 Routine Calibration
    - 10.3.4 GC-FID-ECD-PID System Performance Criteria
  - 10.4 Analytical Procedures
    - 10.4.1 Canister Receipt
    - 10.4.2 GC-MS-SCAN Analysis (With Optional FID System)
    - 10.4.3 GC-MS-SIM Analysis (With Optional FID System)
    - 10.4.4 GC-FID-ECD Analysis (With Optional PID System)

## OUTLINE (Cont.)

11. Cleaning and Certification Program
  - 11.1 Canister Cleaning and Certification
  - 11.2 Sampling System Cleaning and Certification
    - 11.2.1 Cleaning Sampling System Components
    - 11.2.2 Humid Zero Air Certification
    - 11.2.3 Sampler System Certification With Humid Calibration Gas Standards
12. Performance Criteria and Quality Assurance
  - 12.1 Standard Operating Procedures (SOPs)
  - 12.2 Method Relative Accuracy and Linearity
  - 12.3 Method Modification
    - 12.3.1 Sampling
    - 12.3.2 Analysis
  - 12.4 Method Safety
  - 12.5 Quality Assurance
    - 12.5.1 Sampling System
    - 12.5.2 GC-MS-SCAN-SIM System Performance Criteria
    - 12.5.3 GC-Multidetector System Performance Criteria

13. Acknowledgements

14. References

- APPENDIX A - Availability of Audit Cylinders from U.S. Environmental Protection Agency (USEPA) to USEPA Program/Regional Offices, State/Local Agencies and Their Contractors
- APPENDIX B - Operating Procedures for a Portable Gas Chromatograph Equipped With a Photoionization Detector
- APPENDIX C - Installation and Operating Procedures for Alternative Air Toxics Samplers

## METHOD T014

### DETERMINATION OF VOLATILE ORGANIC COMPOUNDS (VOCs) IN AMBIENT AIR USING SUMMA® PASSIVATED CANISTER SAMPLING AND GAS CHROMATOGRAPHIC ANALYSIS

#### 1. Scope

- 1.1 This document describes a procedure for sampling and analysis of volatile organic compounds (VOCs) in ambient air. The method is based on collection of whole air samples in SUMMA® passivated stainless steel canisters. The VOCs are subsequently separated by gas chromatography and measured by mass-selective detector or multidetector techniques. This method presents procedures for sampling into canisters to final pressures both above and below atmospheric pressure (respectively referred to as pressurized and subatmospheric pressure sampling).
- 1.2 This method is applicable to specific VOCs that have been tested and determined to be stable when stored in pressurized and sub-atmospheric pressure canisters. Numerous compounds, many of which are chlorinated VOCs, have been successfully tested for storage stability in pressurized canisters (1,2). However, minimal documentation is currently available demonstrating stability of VOCs in subatmospheric pressure canisters.
- 1.3 The organic compounds that have been successfully collected in pressurized canisters by this method are listed in Table 1. These compounds have been successfully measured at the parts per billion by volume (ppbv) level.

#### 2. Applicable Documents

##### 2.1 ASTM Standards

- D1356 - Definition of Terms Related to Atmospheric Sampling and Analysis
- E260 - Recommended Practice for General Gas Chromatography Procedures
- E355 - Practice for Gas Chromatography Terms and Relationships

##### 2.2 Other Documents

- U.S. Environmental Protection Agency Technical Assistance Document (3) Laboratory and Ambient Air Studies (4-17)

### 3. Summary of Method

- 3.1 Both subatmospheric pressure and pressurized sampling modes use an initially evacuated canister and a pump-ventilated sample line during sample collection. Pressurized sampling requires an additional pump to provide positive pressure to the sample canister. A sample of ambient air is drawn through a sampling train comprised of components that regulate the rate and duration of sampling into a pre-evacuated SUMMA<sup>®</sup> passivated canister.
- 3.2 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 a predetermined laboratory for analysis.
- 3.3 Upon receipt at the laboratory, the canister tag data is recorded and the canister is attached to the analytical system. During analysis, water vapor is reduced in the gas stream by a Nafion<sup>®</sup> dryer (if applicable), and the VOCs are then concentrated by collection in a cryogenically-cooled trap. The cryogen is then removed and the temperature of the trap is raised. The VOCs originally collected in the trap are revolatilized, separated on a GC column, then detected by one or more detectors for identification and quantitation.
- 3.4 The analytical strategy for Method T014 involves using a high-resolution gas chromatograph (GC) coupled to one or more appropriate GC detectors. Historically, detectors for a GC have been divided into two groups: non-specific detectors and specific detectors. The non-specific detectors include, but are not limited to, the nitrogen-phosphorus detector (NPD), the flame ionization detector (FID), the electron capture detector (ECD) and the photoionization detector (PID). The specific detectors include the mass spectrometer (MS) operating in either the selected ion monitoring (SIM) mode or the SCAN mode, or the ion trap detector. The use of these detectors or a combination of these detectors as part of an analytical scheme is determined by the required specificity and sensitivity of the application. While the non-specific detectors are less expensive per analysis and in some cases more sensitive than the specific detector, they vary in specificity and sensitivity for a specific class of compounds. For instance, if multiple halogenated compounds are targeted,

an ECD is usually chosen; if only compounds containing nitrogen or phosphorus are of interest, a NPD can be used; or, if a variety of hydrocarbon compounds are sought, the broad response of the FID or PID is appropriate. In each of these cases, however, the specific identification of the compound within the class is determined only by its retention time, which can be subject to shifts or to interference from other nontargeted compounds. When misidentification occurs, the error is generally a result of a cluttered chromatogram, making peak assignment difficult. In particular, the more volatile organics (chloroethanes, ethyltoluenes, dichlorobenzenes, and various freons) exhibit less well defined chromatographic peaks, leading to misidentification using non-specific detectors. Quantitative comparisons indicate that the FID is more subject to error than the ECD because the ECD is a much more selective detector for a smaller class of compounds which exhibits a stronger response. Identification errors, however, can be reduced by: (a) employing simultaneous detection by different detectors or (b) correlating retention times from different GC columns for confirmation. In either case, interferences on the non-specific detectors can still cause error in identifying a complex sample. The non-specific detector system (GC-NPD-FID-ECD-PID), however, has been used for approximate quantitation of relatively clean samples. The nonspecific detector system can provide a "snapshot" of the constituents in the sample, allowing determination of:

- Extent of misidentification due to overlapping peaks,
- Position of the VOCs within or not within the concentration range of anticipated further analysis by specific detectors (GC-MS-SCAN-SIM) (if not, the sample is further diluted), and
- Existence of unexpected peaks which need further identification by specific detectors.

On the other hand, the use of specific detectors (MS coupled to a GC) allows positive compound identification, thus lending itself to more specificity than the multidetector GC. Operating in the SIM mode, the MS can readily approach the same sensitivity as the

multidetector system, but its flexibility is limited. For SIM operation, the MS is programmed to acquire data for a limited number of targeted compounds while disregarding other acquired information. In the SCAN mode, however, the MS becomes a universal detector, often detecting compounds which are not detected by the multidetector approach. The GC-MS-SCAN will provide positive identification, while the GC-MS-SIM procedure provides quantitation of a restricted "target compound" list of VOCs.

The analyst often must decide whether to use specific or non-specific detectors by considering such factors as project objectives, desired detection limits, equipment availability, cost and personnel capability in developing an analytical strategy. A list of some of the advantages and disadvantages associated with non-specific and specific detectors may assist the analyst in the decision-making process.

#### Non-Specific Multidetector Analytical System

##### Advantages

- o Somewhat lower equipment cost than GC-MS
- o Less sample volume required for analysis
- o More sensitive
  - ECD may be 1000 times more sensitive than GC-MS

##### Disadvantages

- o Multiple detectors to calibrate
- o Compound identification not positive
- o Lengthy data interpretation (one hour each for analysis and data reduction)
- o Interference(s) from co-eluting compound(s)
- o Cannot identify unknown compounds
  - outside range of calibration
  - without standards
- o Does not differentiate targeted compounds from interfering compounds

Specific Detector Analytical System

## GC-MS-SIM

Advantages

- o positive compound identification
- o greater sensitivity than GC-MS-SCAN
- o less operator interpretation than for multidetector GC
- o can resolve co-eluting peaks
- o more specific than the multidetector GC

Disadvantages

- o can't identify non-specified compounds (ions)
- o somewhat greater equipment cost than multidetector GC
- o greater sample volume required than for multidetector GC
- o universality of detector sacrificed to achieve enhancement in sensitivity

## GC-MS-SCAN

- o positive compound identification
- o can identify all compounds
- o less operator interpretation
- o can resolve co-eluting peaks

- o lower sensitivity than GC-MS-SIM
- o greater sample volume required than for multidetector GC
- o somewhat greater equipment cost than multidetector GC

The analytical finish for the measurement chosen by the analyst should provide a definitive identification and a precise quantitation of volatile organics. In a large part, the actual approach to these two objectives is subject to equipment availability. Figure 1 indicates some of the favorite options that are used as an analytical finish. The GC-MS-SCAN option uses a capillary column GC coupled to a MS operated in a scanning mode and supported by spectral library search routines. This option offers the nearest approximation to unambiguous identification and covers a wide range of compounds as defined by the completeness of the spectral library. GC-MS-SIM mode is limited to a set of target compounds which are user defined and is more sensitive than GC-MS-SCAN by virtue of the longer dwell times at the restricted number of m/z values. Both these techniques, but especially the GC-MS-SIM option, can use a supplemental general non-specific detector to verify/identify the presence of VOCs. Finally, the option labelled GC-multidetector system uses a

combination of retention time and multiple general detector verification to identify compounds. However, interference due to nearly identical retention times can affect system quantitation when using this option.

Due to the low concentrations of VOCs encountered in urban air (typically less than 4 ppbv and the majority below 1 ppbv) along with their complicated chromatograms, Method T0-14 strongly recommends the specific detectors (GC-MS-SCAN-SIM) for positive identification and for primary quantitation to ensure that high-quality ambient data is acquired.

For the experienced analyst whose analytical system is limited to the non-specific detectors, Section 10.3 does provide guidelines and example chromatograms showing typical retention times and calibration response factors, and utilizing the non-specific detectors (GC-FID-ECD-PID) analytical system as the primary quantitative technique.

#### 4. Significance

- 4.1 VOCs enter the atmosphere from a variety of sources, including petroleum refineries, synthetic organic chemical plants, natural gas processing plants, and automobile exhaust. Many of these VOCs are acutely toxic; therefore, their determination in ambient air is necessary to assess human health impacts.
- 4.2 Conventional methods for VOC determination use solid sorbent sampling techniques. The most widely used solid sorbent is Tenax<sup>®</sup>. An air sample is drawn through a Tenax<sup>®</sup>-filled cartridge where certain VOCs are trapped on the polymer. The sample cartridge is transferred to a laboratory and analyzed by GC-MS.
- 4.3 VOCs can also be successfully collected in stainless steel canisters. Collection of ambient air samples in canisters provides (1) convenient integration of ambient samples over a specific time period, (e.g., 24 hours); (2) remote sampling and central analysis; (3) ease of storing and shipping samples; (4) unattended sample collection; (5) analysis of samples from multiple sites with one analytical system; and (6) collection of sufficient sample volume to allow assessment of measurement precision and/or analysis of

samples by several analytical systems. However, care must be exercised in selecting, cleaning, and handling sample canisters and sampling apparatus to avoid losses or contamination of the samples. Contamination is a critical issue with canister-based sampling because the canister is the last element in the sampling train.

- 4.4 Interior surfaces of the canisters are treated by the SUMMA<sup>®</sup> passivation process, in which a pure chrome-nickel oxide is formed on the surface. This type of vessel has been used in the past for sample collection and has demonstrated sample storage stability of many specific organic compounds.
- 4.5 This method can be applied to sampling and analysis of not only VOCs, but also some selected semivolatile organic compounds (SVOCs). The term "semivolatile organic compounds" is used to broadly describe organic compounds that are too volatile to be collected by filtration air sampling but not volatile enough for thermal desorption from solid sorbents. SVOCs can generally be classified as those with saturation vapor pressures at 25°C between  $10^{-1}$  and  $10^{-7}$  mm Hg. VOCs are generally classified as those organics having saturated vapor pressures at 25°C greater than  $10^{-1}$  mm Hg.

## 5. Definitions

Note: Definitions used in this document and in any user-prepared Standard Operating Procedures (SOPs) should be consistent with ASTM Methods D1356, E260, and E355. All abbreviations and symbols within this method are defined at point of use.

- 5.1 Absolute canister pressure =  $P_g + P_a$ , where  $P_g$  = gauge pressure in the canister (kPa, psi) and  $P_a$  = barometric pressure (see 5.2).
- 5.2 Absolute pressure - Pressure measured with reference to absolute zero pressure (as opposed to atmospheric pressure), usually expressed as kPa, mm Hg or psia.
- 5.3 Cryogen - A refrigerant used to obtain very low temperatures in the cryogenic trap of the analytical system. A typical cryogen is liquid oxygen (bp -183.0°C) or liquid argon (bp -185.7°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 in a manner very similar to the normal sampling or analytical process.
- 5.5 Gauge pressure - Pressure measured above ambient atmospheric pressure (as opposed to absolute pressure). Zero gauge pressure is equal to ambient atmospheric (barometric) pressure.
- 5.6 MS-SCAN - The GC is coupled to a MS programmed in the SCAN mode to scan all ions repeatedly during the GC run. As used in the current context, this procedure serves as a qualitative identification and characterization of the sample.
- 5.7 MS-SIM - The GC is coupled to a MS programmed to acquire data for only specified ions and to disregard all others. This is performed using SIM coupled to retention time discriminators. The GC-SIM analysis provides quantitative results for selected constituents of the sample gas as programmed by the user.
- 5.8 Megabore® column - Chromatographic column having an internal diameter (I.D.) greater than 0.50 mm. The Megabore® column is a trademark of the J&W Scientific Co. For purposes of this method, Megabore® refers to chromatographic columns with 0.53 mm I.D.
- 5.9 Pressurized sampling - Collection of an air sample in a canister with a (final) canister pressure above atmospheric pressure, using a sample pump.
- 5.10 Qualitative accuracy - The ability of an analytical system to correctly identify compounds.
- 5.11 Quantitative accuracy - The ability of an analytical system to correctly measure the concentration of an identified compound.
- 5.12 Static calibration - Calibration of an analytical system using standards in a form different than the samples to be analyzed. An example of a static calibration would be injecting a small volume of a high concentration standard directly onto a GC column, bypassing the sample extraction and preconcentration portion of the analytical system.

5.13 Subatmospheric sampling - Collection of an air sample in an evacuated canister at a (final) canister pressure below atmospheric pressure, without the assistance of a sampling pump. The canister is filled as the internal canister pressure increases to ambient or near ambient pressure. An auxiliary vacuum pump may be used as part of the sampling system to flush the inlet tubing prior to or during sample collection.

## 6. Interferences and Limitations

- 6.1 Interferences can occur in sample analysis if moisture accumulates in the dryer (see Section 10.1.1.2). An automated cleanup procedure that periodically heats the dryer to about 100°C while purging with zero air eliminates any moisture buildup. This procedure does not degrade sample integrity.
- 6.2 Contamination may occur in the sampling system if canisters are not properly cleaned before use. Additionally, all other sampling equipment (e.g., pump and flow controllers) should be thoroughly cleaned to ensure that the filling apparatus will not contaminate samples. Instructions for cleaning the canisters and certifying the field sampling system are described in Sections 12.1 and 12.2, respectively.
- 6.3 Because the GC-MS analytical system employs a Nafion® permeable membrane dryer to remove water vapor selectively from the sample stream, polar organic compounds may permeate concurrent with the moisture molecule. Consequently, the analyst should quantitate his or her system with the specific organic constituents under examination.

## 7. Apparatus

### 7.1 Sample Collection

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

- 7.1.1 Subatmospheric Pressure (See Figure 2 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 SUMMA® passivated interior surfaces (Scientific Instrumentation Specialists, Inc., P.O. Box 8941, Moscow, ID 83843, or Anderson Samplers, Inc., 4215-C Wendell Dr., Atlanta, GA, 30336, or equivalent).
  - 7.1.1.3 Stainless steel vacuum/pressure gauge - 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 (Matheson, P.O. Box 136, Morrow, GA 30200, Model 63-3704, or equivalent). Gauges should be tested clean and leak tight.
  - 7.1.1.4 Electronic mass flow controller - capable of maintaining a constant flow rate ( $\pm 10\%$ ) over a sampling period of up to 24 hours and under conditions of changing temperature (20-40°C) and humidity (Tylan Corp., 19220 S. Normandie Ave., Torrance, CA 90502, Model FC-260, or equivalent).
  - 7.1.1.5 Particulate matter filter - 2-um sintered stainless steel in-line filter (Nupro Co., 4800 E. 345th St., Willoughby, OH 44094, Model SS-2F-K4-2, or equivalent).
  - 7.1.1.6 Electronic timer - for unattended sample collection (Paragon Elect. Co., 606 Parkway Blvd., P.O. Box 28, Twin Rivers, WI 54201, Model 7008-00, or equivalent).
  - 7.1.1.7 Solenoid valve - electrically-operated, bi-stable solenoid valve (Skinner Magnelatch Valve, New Britain, CT, Model V5RAM49710, or equivalent) with Viton® seat and o-rings.
  - 7.1.1.8 Chromatographic grade stainless steel tubing and fittings - for interconnections (Alltech Associates, 2051 Waukegan Rd., Deerfield, IL 60015, Cat. #8125,

- or equivalent). All such materials in contact with sample, analyte, and support gases prior to analysis should be chromatographic grade stainless steel.
- 7.1.1.9 Thermostatically controlled heater - to maintain temperature inside insulated sampler enclosure above ambient temperature (Watlow Co., Pfafftown, NC, Part 04010080, or equivalent).
  - 7.1.1.10 Heater thermostat - automatically regulates heater temperature (Elmwood Sensors, Inc., 500 Narragansett Park Dr., Pawtucket RI 02861, Model 3455-RC-0100-0222, or equivalent).
  - 7.1.1.11 Fan - for cooling sampling system (EG&G Rotron, Woodstock, NY, Model SUZAI, or equivalent).
  - 7.1.1.12 Fan thermostat - automatically regulates fan operation (Elmwood Sensors, Inc., Pawtucket, RI, Model 3455-RC-0100-0244, or equivalent).
  - 7.1.1.13 Maximum-minimum thermometer - records highest and lowest temperatures during sampling period (Thomas Scientific, Brooklyn Thermometer Co., Inc., P/N 9327H30, or equivalent).
  - 7.1.1.14 Nupro stainless steel shut-off valve - leak free, for vacuum/pressure gauge.
  - 7.1.1.15 Auxiliary vacuum pump - continuously draws ambient air to be sampled 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 (Conrac, Cramer Div., Old Saybrook, CT, Type 6364, P/N 10082, or equivalent).
  - 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

## 7.1.2 Pressurized (Figure 2 With Metal Bellows Type Pump and Figure 3)

7.1.2.1 Sample pump - stainless steel, metal bellows type (Metal Bellows Corp., 1075 Providence Highway, Sharon, MA 02067, Model MB-151, or equivalent), 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 Center (18,19) and is illustrated in Figure 3. This flow system uses, in order, a pump, a mechanical flow regulator, and a mechanical compensating 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. Interferences using this configuration have been minimal.]

7.1.2.2 Other supporting materials - all other components of the pressurized sampling system (Figure 2 with metal bellows type pump and Figure 3) are similar to components discussed in Sections 7.1.1.1 through 7.1.1.16.

## 7.2 Sample Analysis

### 7.2.1 GC-MS-SCAN Analytical System (See Figure 4)

7.2.1.1 The GC-MS-SCAN analytical system must be capable of acquiring and processing data in the MS-SCAN mode.

7.2.1.2 Gas chromatograph - capable of sub-ambient temperature programming for the oven, with other generally standard features such as gas flow regulators, automatic control of valves and integrator, etc. Flame ionization detector optional. (Hewlett Packard, Rt. 41, Avondale, PA 19311, Model 5880A, with oven temperature control and Level 4 BASIC programming, or equivalent.)

7.2.1.3 Chromatographic detector - mass-selective detector (Hewlett Packard, 3000-T Hanover St., 9B, Palo Alto, CA 94304, Model HP-5970 MS, or equivalent), equipped with computer and appropriate software (Hewlett Packard, 3000-T Hanover St., 9B, Palo Alto, CA 94304,

HP-216 Computer, Quicksilver MS software, Pascal 3.0, mass storage 9133 HP Winchester with 3.5 inch floppy disk, or equivalent). The GC-MS is set in the SCAN mode, where the MS screens the sample for identification and quantitation of VOC species.

- 7.2.1.4 Cryogenic trap with temperature control assembly - refer to Section 10.1.1.3 for complete description of trap and temperature control assembly (Nutech Corporation, 2142 Geer St., Durham, NC, 27704, Model 320-01, or equivalent).
- 7.2.1.5 Electronic mass flow controllers (3) - maintain constant flow (for carrier gas and sample gas) and to provide analog output to monitor flow anomalies (Tylan Model 260, 0-100 cm<sup>3</sup>/min, or equivalent).
- 7.2.1.6 Vacuum pump - general purpose laboratory pump, capable of drawing the desired sample volume through the cryogenic trap (Thomas Industries, Inc., Sheboygan, WI, Model 107BA20, or equivalent).
- 7.2.1.7 Chromatographic grade stainless steel tubing and stainless steel plumbing fittings - refer to Section 7.1.1.8 for description.
- 7.2.1.8 Chromatographic column - to provide compound separation such as shown in Table 5 (Hewlett Packard, Rt. 41, Avondale, PA 19311, OV-1 capillary column, 0.32 mm x 50 m with 0.88 um crosslinked methyl silicone coating, or equivalent).
- 7.2.1.9 Stainless steel vacuum/pressure gauge (optional) - capable of measuring vacuum (-101.3 to 0 kPa) and pressure (0-206 kPa) in the sampling system (Matheson, P.O. Box 136, Morrow, GA 30200, Model 63-3704, or equivalent). Gauges should be tested clean and leak tight.
- 7.2.1.10 Stainless steel cylinder pressure regulators - standard two-stage cylinder regulators with pressure gauges for helium, zero air and hydrogen gas cylinders.
- 7.2.1.11 Gas purifiers (3) - used to remove organic impurities and moisture from gas streams (Hewlett Packard, Rt. 41, Avondale, PA, 19311, P/N 19362 - 60500, or equivalent).

- 7.2.1.12 Low dead-volume tee (optional) - used to split the exit flow from the GC column (Alltech Associates, 2051 Waukegan Rd., Deerfield, IL 60015, Cat. #5839, or equivalent).
  - 7.2.1.13 Nafion® dryer - consisting of Nafion tubing co-axially mounted within larger tubing (Perma Pure Products, 8 Executive Drive, Toms River, NJ, 08753, Model MD-125-48, or equivalent). Refer to Section 10.1.1.2 for description.
  - 7.2.1.14 Six-port gas chromatographic valve - (Seismograph Service Corp, Tulsa, OK, Seiscor Model VIII, or equivalent).
  - 7.2.1.15 Chart recorder (optional) - compatible with the detector output signals to record optional FID detector response to the sample.
  - 7.2.1.16 Electronic integrator (optional) - compatible with the detector output signal of the FID and capable of integrating the area of one or more response peaks and calculating peak areas corrected for baseline drift.
- 7.2.2 GC-MS-SIM Analytical System (See Figure 4)
- 7.2.2.1 The GC-MS-SIM analytical system must be capable of acquiring and processing data in the MS-SIM mode.
  - 7.2.2.2 All components of the GC-MS-SIM system are identical to Sections 7.2.1.2 through 7.2.1.16.
- 7.2.3 GC-Multidetector Analytical System (See Figure 5 and Figure 6)
- 7.2.3.1 Gas chromatograph with flame ionization and electron capture detectors (photoionization detector optional) - capable of sub-ambient temperature programming for the oven and simultaneous operation of all detectors, and with other generally standard features such as gas flow regulators, automatic control of valves and integrator, etc. (Hewlett Packard, Rt. 41, Avondale, PA 19311, Model 5880A, with oven temperature control and Level 4 BASIC programming, or equivalent).

- 7.2.3.2 Chart recorders - compatible with the detector output signals to record detector response to the sample.
- 7.2.3.3 Electronic integrator - compatible with the detector output signals and capable of integrating the area of one or more response peaks and calculating peak areas corrected for baseline drift.
- 7.2.3.4 Six-port gas chromatographic valve - (Seismograph Service Corp, Tulsa, OK, Seiscor Model VIII, or equivalent)
- 7.2.3.5 Cryogenic trap with temperature control assembly - refer to Section 10.1.1.3 for complete description of trap and temperature control assembly (Nutech Corporation, 2142 Geer St., Durham, NC 27704, Model 320-01, or equivalent).
- 7.2.3.6 Electronic mass flow controllers (3) - maintain constant flow (for carrier gas, nitrogen make-up gas and sample gas) and to provide analog output to monitor flow anomalies (Tylan Model 260, 0-100 cm<sup>3</sup>/min, or equivalent).
- 7.2.3.7 Vacuum pump - general purpose laboratory pump, capable of drawing the desired sample volume through the cryogenic trap (see 7.2.1.6 for source and description).
- 7.2.3.8 Chromatographic grade stainless steel tubing and stainless steel plumbing fittings - refer to Section 7.1.1.8 for description.
- 7.2.3.9 Chromatographic column - to provide compound separation such as shown in Table 7. (Hewlett Packard, Rt. 41, Avondale, PA 19311, OV-1 capillary column, 0.32 mm x 50 m with 0.88 um crosslinked methyl silicone coating, or equivalent). [Note: Other columns (e.g., DB-624) can be used as long as the system meets user needs. The wider Megabore<sup>®</sup> column (i.e., 0.53 mm I.D.) is less susceptible to plugging as a result of trapped water, thus eliminating the need for a Nafion<sup>®</sup> dryer in the analytical system. The Megabore<sup>®</sup> column has sample capacity approaching that of a packed column, while retaining much of the peak resolution traits of narrower columns (i.e., 0.32 mm I.D.).

- 7.2.3.10 Vacuum/pressure gauges (3) - refer to Section 7.2.1.9 for description.
- 7.2.3.11 Cylinder pressure stainless steel regulators - standard, two-stage cylinder regulators with pressure gauges for helium, zero air, nitrogen, and hydrogen gas cylinders.
- 7.2.3.12 Gas purifiers (4) - used to remove organic impurities and moisture from gas streams (Hewlett-Packard, Rt. 41, Avondale, PA, 19311, P/N 19362 - 60500, or equivalent).
- 7.2.3.13 Low dead-volume tee - used to split (50/50) the exit flow from the GC column (Alltech Associates, 2051 Waukegan Rd., Deerfield, IL 60015, Cat. #5839, or equivalent).

### 7.3 Canister Cleaning System (See Figure 7)

- 7.3.1 Vacuum pump - capable of evacuating sample canister(s) to an absolute pressure of <0.05 mm Hg.
- 7.3.2 Manifold - stainless steel manifold with connections for simultaneously cleaning several canisters.
- 7.3.3 Shut-off valve(s) - seven (7) on-off toggle valves.
- 7.3.4 Stainless steel vacuum gauge - capable of measuring vacuum in the manifold to an absolute pressure of 0.05 mm Hg or less.
- 7.3.5 Cryogenic trap (2 required) - stainless steel U-shaped open tubular trap cooled with liquid oxygen or argon to prevent contamination from back diffusion of oil from vacuum pump and to provide clean, zero air to sample canister(s).
- 7.3.6 Stainless steel pressure gauges (2) - 0-345 kPa (0-50 psig) to monitor zero air pressure.
- 7.3.7 Stainless steel flow control valve - to regulate flow of zero air into canister(s).
- 7.3.8 Humidifier - pressurizable water bubbler containing high performance liquid chromatography (HPLC) grade deionized water or other system capable of providing moisture to the zero air supply.
- 7.3.9 Isothermal oven (optional) for heating canisters (Fisher Scientific, Pittsburgh, PA, Model 349, or equivalent).

## 7.4 Calibration System and Manifold (See Figure 8)

- 7.4.1 Calibration manifold - glass manifold, (1.25 cm I.D. x 66 cm) with sampling ports and internal baffles for flow disturbance to ensure proper mixing.
- 7.4.2 Humidifier - 500-mL impinger flask containing HPLC grade deionized water.
- 7.4.3 Electronic mass flow controllers - one 0 to 5 L/min and one 0 to 50 cm<sup>3</sup>/min (Tylan Corporation, 23301-TS Wilmington Ave., Carson, CA, 90745, Model 2160, or equivalent).
- 7.4.4 Teflon® filter(s) - 47-mm Teflon® filter for particulate control, best source.

## 8. Reagents and Materials

- 8.1 Gas cylinders of helium, hydrogen, nitrogen, and zero air - ultrahigh purity grade, best source.
- 8.2 Gas calibration standards - cylinder(s) containing approximately 10 ppmv of each of the following compounds of interest:

vinyl chloride	1,2-dibromoethane
vinylidene chloride	tetrachloroethylene
1,1,2-trichloro-1,2,2-trifluoroethane	chlorobenzene
chloroform	benzyl chloride
1,2-dichloroethane	hexachloro-1,3-butadiene
benzene	methyl chloroform
toluene	carbon tetrachloride
Freon 12	trichloroethylene
methyl chloride	cis-1,3-dichloropropene
1,2-dichloro-1,1,2,2-tetrafluoroethane	trans-1,3-dichloropropene
methyl bromide	ethylbenzene
ethyl chloride	o-xylene
Freon 11	m-xylene
dichloromethane	p-xylene
1,1-dichloroethane	styrene
cis-1,2-dichloroethylene	1,1,2,2-tetrachloroethane
1,2-dichloropropane	1,3,5-trimethylbenzene
1,1,2-trichloroethane	1,2,4-trimethylbenzene
	m-dichlorobenzene
	o-dichlorobenzene
	p-dichlorobenzene
	1,2,4-trichlorobenzene

The cylinder(s) should be traceable to a National Bureau of Standards (NBS) Standard Reference Material (SRM) or to a NBS/EPA approved Certified Reference Material (CRM). The components may be purchased in one cylinder or may be separated into different cylinders. Refer to manufacturer's specification for guidance on purchasing and mixing VOCs in gas cylinders. Those compounds purchased should match one's own target list.

- 8.3 Cryogen - liquid oxygen (bp  $-183.0^{\circ}\text{C}$ ), or liquid argon (bp  $-185.7^{\circ}\text{C}$ ), best source.
- 8.4 Gas purifiers - connected in-line between hydrogen, nitrogen, and zero air gas cylinders and system inlet line, to remove moisture and organic impurities from gas streams (Alltech Associates, 2051 Waukegan Road, Deerfield, IL, 60015, or equivalent).
- 8.5 Deionized water - high performance liquid chromatography (HPLC) grade, ultrahigh purity (for humidifier), best source.
- 8.6 4-bromofluorobenzene - used for tuning GC-MS, best source.
- 8.7 Hexane - for cleaning sampling system components, reagent grade, best source.
- 8.8 Methanol - for cleaning sampling system components, reagent grade, best source.

## 9. Sampling System

### 9.1 System Description

#### 9.1.1 Subatmospheric Pressure Sampling [See Figure 2 (Without Metal Bellows Type Pump)]

9.1.1.1 In preparation for subatmospheric sample collection in a canister, the canister is evacuated to 0.05 mm Hg. When 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-integrated samples (duration of 12 to 24 hours) taken through a flow-restrictive inlet (e.g., mass flow controller, critical orifice).

9.1.1.2 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.

### 9.1.2 Pressurized Sampling [See Figure 2 (With Metal Bellows Type Pump)]

9.1.2.1 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 103-206 kPa (15-30 psig) final canister pressure. For example, a 6-liter evacuated canister can be filled at 10 cm<sup>3</sup>/min for 24 hours to achieve a final pressure of about 144 kPa (21 psig).

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

### 9.1.3 All Samplers

9.1.3.1 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 (cm<sup>3</sup>/min).

P = final canister pressure, atmospheres absolute. P is approximately equal to

$$\frac{\text{kPa}_{\text{gauge}} + 1}{101.2}$$

V = volume of the canister (cm<sup>3</sup>).

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

$$F = \frac{2 \times 6000}{24 \times 60} = 8.3 \text{ cm}^3/\text{min}$$

- 9.1.3.2 For automatic operation, the timer is wired 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 close the valve when stopping the pump.
- 9.1.3.3 The use of the Skinner Magnelatch valve 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 9(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 9(b).

- 9.1.3.4 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. If a critical orifice is used, some drop in the flow rate may occur near the end of the sample period as the canister pressure approaches the final calculated pressure.
- 9.1.3.5 As an option, a second electronic timer (see Section 7.1.1.6) may be used to start the auxiliary pump several hours prior to the sampling period to flush and condition the inlet line.
- 9.1.3.6 Prior to field use, each sampling system must pass a humid zero air certification (see Section 12.2.2). All plumbing should be checked carefully for leaks. The canisters must also pass a humid zero air certification before use (see Section 12.1).

## 9.2 Sampling Procedure

- 9.2.1 The sample canister should be cleaned and tested according to the procedure in Section 12.1.
- 9.2.2 A sample collection system is assembled as shown in Figure 2 (and Figure 3) and must meet certification requirements as outlined in Section 12.2.3. [Note: The sampling system should be contained in an appropriate enclosure.]
- 9.2.3 Prior to locating the sampling system, the user may want to perform "screening analyses" using a portable GC system, as outlined in Appendix B, 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.
- 9.2.4 After "screening analysis," the sampling system is located. Temperatures of ambient air and sampler box interior are recorded on canister sampling field data sheet (Figure 10). [Note: The following discussion is related to Figure 2.]

- 9.2.5 To verify correct sample flow, a "practice" (evacuated) canister is used in the sampling system. [Note: For a subatmospheric sampler, the 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  $\pm 10\%$ . 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 two 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  $\text{cm}^3/\text{min}$  for 24 hr, 7.0  $\text{cm}^3/\text{min}$  for 12 hr). Record final flow under "CANISTER FLOW RATE," Figure 10.
- 9.2.6 The sampler is turned off and the elapsed time meter is reset to 000.0. Note: Any time the sampler is turned off, wait at least 30 seconds to turn the sampler back on.
- 9.2.7 The "practice" canister and certified mass flow meter are disconnected and a clean certified (see Section 12.1) canister is attached to the system.
- 9.2.8 The canister valve and vacuum/pressure gauge valve are opened.
- 9.2.9 Pressure/vacuum in the canister is recorded on the canister sampling field data sheet (Figure 10) as indicated by the sampler vacuum/pressure gauge.
- 9.2.10 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 sampling field data sheet.
- 9.2.11 The electronic timer is set to begin and stop the sampling period at the appropriate times. Sampling commences and stops by the programmed electronic timer.

- 9.2.12 After the desired sampling period, the maximum, minimum, current interior temperature and current ambient temperature are recorded on the sampling field data sheet. The current reading from the flow controller is recorded.
- 9.2.13 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 sampling field data sheet. 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.
- 9.2.14 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 Magelatch valve of the sampling system. The final flow rate is recorded on the canister sampling field data sheet (see Figure 10). [Note: For a pressurized system, the final flow may be measured directly.] The sampler is turned off.
- 9.2.15 An identification tag is attached to the canister. Canister serial number, sample number, location, and date are recorded on the tag.

## 10. Analytical System (See Figures 4, 5 and 6)

### 10.1 System Description

#### 10.1.1 GC-MS-SCAN System

- 10.1.1.1 The analytical system is comprised of a GC equipped with a mass-selective detector set in the SCAN mode (see Figure 4). All ions are scanned by the MS repeatedly during the

GC run. The system includes a computer and appropriate software for data acquisition, data reduction, and data reporting. A 400 cm<sup>3</sup> air sample is collected from the canister into the analytical system. The sample air is first passed through a Nafion<sup>®</sup> dryer, through the 6-port chromatographic valve, then routed into a cryogenic trap. [Note: While the GC-multidetector analytical system does not employ a Nafion<sup>®</sup> dryer for drying the sample gas stream, it is used here because the GC-MS system utilizes a larger sample volume and is far more sensitive to excessive moisture than the GC-multidetector analytical system. Moisture can adversely affect detector precision. The Nafion<sup>®</sup> dryer also prevents freezing of moisture on the 0.32 mm I.D. column, which may cause column blockage and possible breakage.] The trap is heated (-160°C to 120°C in 60 sec) and the analyte is injected onto the OV-1 capillary column (0.32 mm x 50 m). [Note: Rapid heating of the trap provides efficient transfer of the sample components onto the gas chromatographic column.] Upon sample injection onto the column, the MS computer is signaled by the GC computer to begin detection of compounds which elute from the column. The gas stream from the GC is scanned within a preselected range of atomic mass units (amu). For detection of compounds in Table 1, the range should be 18 to 250 amu, resulting in a 1.5 Hz repetition rate. Six (6) scans per eluting chromatographic peak are provided at this rate. The 10-15 largest peaks are chosen by an automated data reduction program, the three scans nearest the peak apex are averaged, and a background subtraction is performed. A library search is then performed and the top ten best matches for each peak are listed. A qualitative characterization

of the sample is provided by this procedure. A typical chromatogram of VOCs determined by GC-MS-SCAN is illustrated in Figure 11(a).

10.1.1.2 A Nafion® permeable membrane dryer is used to remove water vapor selectively from the sample stream. The permeable membrane consists of Nafion® tubing (a copolymer of tetrafluoroethylene and fluorosulfonyl monomer) that is coaxially mounted within larger tubing. The sample stream is passed through the interior of the Nafion® tubing, allowing water (and other light, polar compounds) to permeate through the walls into a dry air purge stream flowing through the annular space between the Nafion® and outer tubing. [Note: To prevent excessive moisture build-up and any memory effects in the dryer, a clean-up procedure involving periodic heating of the dryer (100°C for 20 minutes) while purging with dry zero air (500 cm<sup>3</sup>/min) should be implemented as part of the user's SOP manual. The clean-up procedure is repeated during each analysis (see Section 14, reference 7). Recent studies have indicated no substantial loss of targeted VOCs utilizing the above clean-up procedure (7). This cleanup procedure is particularly useful when employing cryogenic preconcentration of VOCs with subsequent GC analysis using a 0.32 mm I.D. column because excess accumulated water can cause trap and column blockage and also adversely affect detector precision. In addition, the improvement in water removal from the sampling stream will allow analyses of much larger volumes of sample air in the event that greater system sensitivity is required for targeted compounds.]

10.1.1.3 The packed metal tubing used for reduced temperature trapping of VOCs is shown in Figure 12. The cooling unit is comprised of a 0.32 cm outside diameter (O.D.) nickel tubing loop packed with 60-80 mesh Pyrex® beads (Nutech Model 320-01, or equivalent). The nickel tubing loop is wound onto a cylindrically formed tube heater (250 watt). A cartridge heater (25 watt) is sandwiched between pieces of aluminum plate at the trap inlet and outlet to provide additional heat to eliminate cold spots in the transfer tubing. During operation, the trap is inside a two-section stainless steel shell which is well insulated. Rapid heating (-150 to +100°C in 55 s) is accomplished by direct thermal contact between the heater and the trap tubing. Cooling is achieved by vaporization of the cryogen. In the shell, efficient cooling (+120 to -150°C in 225 s) is facilitated by confining the vaporized cryogen to the small open volume surrounding the trap assembly. The trap assembly and chromatographic valve are mounted on a baseplate fitted into the injection and auxiliary zones of the GC on an insulated pad directly above the column oven when used with the Hewlett-Packard 5880 GC. [Note: Alternative trap assembly and connection to the GC may be used depending upon user's requirements.] The carrier gas line is connected to the injection end of the analytical column with a zero-dead-volume fitting that is usually held in the heated zone above the GC oven. A 15 cm x 15 cm x 24 cm aluminum box is fitted over the sample handling elements to complete the package. Vaporized cryogen is vented through the top of the box.

10.1.1.4 As an option, the analyst may wish to split the gas stream exiting the column with a low dead-volume tee, passing one-third of the sample gas (1.0 mL/min) to the mass-selective detector and the remaining two-thirds (2.0 mL/min) through a flame ionization detector, as illustrated as an option in Figure 4. The use of the specific detector (MS-SCAN) coupled with the non-specific detector (FID) enables enhancement of data acquired from a single analysis. In particular, the FID provides the user:

- o Semi-real time picture of the progress of the analytical scheme;
- o Confirmation by the concurrent MS analysis of other labs that can provide only FID results; and
- o Ability to compare GC-FID with other analytical laboratories with only GC-FID capability.

## 10.1.2 GC-MS-SIM System

10.1.2.1 The analytical system is comprised of a GC equipped with an OV-1 capillary column (0.32 mm x 50 m) and a mass-selective detector set in the SIM mode (see Figure 4). The GC-MS is set up for automatic, repetitive analysis. The system is programmed to acquire data for only the target compounds and to disregard all others. The sensitivity is 0.1 ppbv for a 250 cm<sup>3</sup> air sample with analytical precision of about 5% relative standard deviation. Concentration of compounds based upon a previously installed calibration table is reported by an automated data reduction program. A Nafion<sup>®</sup> dryer is also employed by this analytical system prior to cryogenic preconcentration; therefore, many polar compounds are not identified by this procedure.

10.1.2.2 SIM analysis is based on a combination of retention times and relative abundances of selected ions (see Table 2). These qualifiers are stored on the hard disk of the GC-MS computer and are applied for identification of each chromatographic peak. The retention time qualifier is determined to be  $\pm 0.10$  minute of the library retention time of the compound. The acceptance level for relative abundance is determined to be  $\pm 15\%$  of the expected abundance, except for vinyl chloride and methylene chloride, which is determined to be  $\pm 25\%$ . Three ions are measured for most of the forty compounds. When compound identification is made by the computer, any peak that fails any of the qualifying tests is flagged (e.g., with an \*). All the data should be manually examined by the analyst to determine the reason for the flag and whether the compound should be reported as found. While this adds some subjective judgment to the analysis, computer-generated identification problems can be clarified by an experienced operator. Manual inspection of the quantitative results should also be performed to verify concentrations outside the expected range. A typical chromatogram of VOCs determined by GC-MS-SIM mode is illustrated in Figure 11(b).

### 10.1.3 GC-Multidetector (GC-FID-ECD) System with Optional PID

10.1.3.1 The analytical system (see Figure 5) is comprised of a gas chromatograph equipped with a capillary column and electron capture and flame ionization detectors (see Figure 5). In typical operation, sample air from pressurized canisters is vented past the inlet to the analytical system from the canister at a flow rate of  $75 \text{ cm}^3/\text{min}$ . For analysis, only

is vented to the atmosphere. Sub-ambient pressure canisters are connected directly to the inlet. The sample gas stream is routed through a six port chromatographic valve and into the cryogenic trap for a total sample volume of 490 cm<sup>3</sup>. [Note: This represents a 14 minute sampling period at a rate of 35 cm<sup>3</sup>/min.] The trap (see Section 10.1.1.3) is cooled to -150°C by controlled release of a cryogen. VOCs and SVOCs are condensed on the trap surface while N<sub>2</sub>, O<sub>2</sub>, and other sample components are passed to the pump. After the organic compounds are concentrated, the valve is switched and the trap is heated. The revolatilized compounds are transported by helium carrier gas at a rate of 4 cm<sup>3</sup>/min to the head of the Megabore® OV-1 capillary column (0.53 mm x 30 m). Since the column initial temperature is at -50°C, the VOCs and SVOCs are cryofocussed on the head of the column. Then, the oven temperature is programmed to increase and the VOCs/SVOCs in the carrier gas are chromatographically separated. The carrier gas containing the separated VOCs/SVOCs is then directed to two parallel detectors at a flow rate of 2 cm<sup>3</sup>/min each. The detectors sense the presence of the speciated VOCs/SVOCs, and the response is recorded by either a strip chart recorder or a data processing unit.

10.1.3.2 Typical chromatograms of VOCs determined by the GC-FID-ECD analytical system are illustrated in Figures 11(c) and 11(d), respectively.

10.1.3.3 Helium is used as the carrier gas (4 cm<sup>3</sup>/min) to purge residual air from the trap at the end of the sampling phase and to carry the revolatilized VOCs through the Megabore® GC column. Moisture and organic impurities are removed from the helium gas stream by a chemical purifier installed in the GC (see

Section 7.2.1.11). After exiting the OV-1 Megabore® column, the carrier gas stream is split to the two detectors at rates of 2 cm<sup>3</sup>/min each.

- 10.1.3.4 Gas scrubbers containing Drierite® or silica gel and 5A molecular sieve are used to remove moisture and organic impurities from the zero air, hydrogen, and nitrogen gas streams. [Note: Purity of gas purifiers is checked prior to use by passing humid zero-air through the gas purifier and analyzing according to Section 12.2.2.]
- 10.1.3.5 All lines should be kept as short as practical. All tubing used for the system should be chromatographic grade stainless steel connected with stainless steel fittings. After assembly, the system should be checked for leaks according to manufacturer's specifications.
- 10.1.3.6 The FID burner air, hydrogen, nitrogen (make-up), and helium (carrier) flow rates should be set according to the manufacturer's instructions to obtain an optimal FID response while maintaining a stable flame throughout the analysis. Typical flow rates are: burner air, 450 cm<sup>3</sup>/min; hydrogen, 30 cm<sup>3</sup>/min; nitrogen, 30 cm<sup>3</sup>/min; helium, 2 cm<sup>3</sup>/min.
- 10.1.3.7 The ECD nitrogen make-up gas and helium carrier flow rates should be set according to manufacturer's instructions to obtain an optimal ECD response. Typical flow rates are: nitrogen, 76 cm<sup>3</sup>/min and helium, 2 cm<sup>3</sup>/min.
- 10.1.3.8 The GC-FID-ECD could be modified to include a PID (see Figure 6) for increased sensitivity (20). In the photoionization process, a molecule is ionized by ultraviolet light as follows:  $R + h\nu \rightarrow R^+ + e^-$ , where R<sup>+</sup> is the ionized species and a photon is represented by hν, with energy less than or equal to the ionization potential of

the molecule. Generally all species with an ionization potential less than the ionization energy of the lamp are detected. Because the ionization potential of all major components of air ( $O_2$ ,  $N_2$ ,  $CO$ ,  $CO_2$ , and  $H_2O$ ) is greater than the ionization energy of lamps in general use, they are not detected. The sensor is comprised of an argon-filled, ultraviolet (UV) light source where a portion of the organic vapors are ionized in the gas stream. A pair of electrodes are contained in a chamber adjacent to the sensor. When a positive potential is applied to the electrodes, any ions formed by the absorption of UV light are driven by the created electronic field to the cathode, and the current (proportional to the organic vapor concentration) is measured. The PID is generally used for compounds having ionization potentials less than the ratings of the ultraviolet lamps. This detector is used for determination of most chlorinated and oxygenated hydrocarbons, aromatic compounds, and high molecular weight aliphatic compounds. Because the PID is insensitive to methane, ethane, carbon monoxide, carbon dioxide, and water vapor, it is an excellent detector. The electron volt rating is applied specifically to the wavelength of the most intense emission line of the lamp's output spectrum. Some compounds with ionization potentials above the lamp rating can still be detected due to the presence of small quantities of more intense light. A typical system configuration associated with the GC-FID-ECD-PID is illustrated in Figure 6. This system is currently being used in EPA's FY-88 Urban Air Toxics Monitoring Program.

## 10.2 GC-MS-SCAN-SIM System Performance Criteria

### 10.2.1 GC-MS System Operation

- 10.2.1.1 Prior to analysis, the GC-MS system is assembled and checked according to manufacturer's instructions.
- 10.2.1.2 Table 3.0 outlines general operating conditions for the GC-MS-SCAN-SIM system with optional FID.
- 10.2.1.3 The GC-MS system is first challenged with humid zero air (see Section 11.2.2).
- 10.2.1.4 The GC-MS and optional FID system is acceptable if it contains less than 0.2 ppbv of targeted VOCs.

### 10.2.2 Daily GC-MS Tuning (See Figure 13)

- 10.2.2.1 At the beginning of each day or prior to a calibration, the GC-MS system must be tuned to verify that acceptable performance criteria are achieved.
- 10.2.2.2 For tuning the GC-MS, a cylinder containing 4-bromofluorobenzene is introduced via a sample loop valve injection system. [Note: Some systems allow auto-tuning to facilitate this process.] The key ions and ion abundance criteria that must be met are illustrated in Table 4. Analysis should not begin until all those criteria are met.
- 10.2.2.3 The GC-MS tuning standard could also be used to assess GC column performance (chromatographic check) and as an internal standard. Obtain a background correction mass spectra of 4-bromofluorobenzene and check that all key ions criteria are met. If the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved.
- 10.2.2.4 The performance criteria must be achieved before any samples, blanks or standards are analyzed. If

any key ion abundance observed for the daily 4-bromofluorobenzene mass tuning check differs by more than 10% absolute abundance from that observed during the previous daily tuning, the instrument must be retuned or the sample and/or calibration gases reanalyzed until the above condition is met.

### 10.2.3 GC-MS Calibration (See Figure 13)

[Note: Initial and routine calibration procedures are illustrated in Figure 13.]

10.2.3.1 Initial Calibration - Initially, a multipoint dynamic calibration (three levels plus humid zero air) is performed on the GC-MS system, before sample analysis, with the assistance of a calibration system (see Figure 8). The calibration system uses NBS traceable standards or NBS/EPA CRMs in pressurized cylinders [containing a mixture of the targeted VOCs at nominal concentrations of 10 ppmv in nitrogen (Section 8.2)] as working standards to be diluted with humid zero air. The contents of the working standard cylinder(s) are metered ( $2 \text{ cm}^3/\text{min}$ ) into the heated mixing chamber where they are mixed with a 2-L/min humidified zero air gas stream to achieve a nominal 10 ppbv per compound calibration mixture (see Figure 8). This nominal 10 ppbv standard mixture is allowed to flow and equilibrate for a minimum of 30 minutes. After the equilibration period, the gas standard mixture is sampled and analyzed by the real-time GC-MS system [see Figure 8(a) and Section 7.2.1]. The results of the analyses are averaged, flow audits are performed on the mass flow meters and the calculated concentration compared to generated values. After the GC-MS is calibrated at three concentration levels, a second humid zero air sample is passed through the system and analyzed. The second humid zero air test is used to verify that the GC-MS system is certified clean (less than 0.2 ppbv of target compounds).

10.2.3.2 As an alternative, a multipoint humid static calibration (three levels plus zero humid air) can be performed on the GC-MS system. During the humid static calibration analyses, three (3) SUMMA<sup>®</sup> passivated canisters are filled each at a different concentration between 1-20 ppbv from the calibration manifold using a pump and mass flow control arrangement [see Figure 8(c)]. The canisters are then delivered to the GC-MS to serve as calibration standards. The canisters are analyzed by the MS in the SIM mode, each analyzed twice. The expected retention time and ion abundance (see Table 2 and Table 5) are used to verify proper operation of the GC-MS system. A calibration response factor is determined for each analyte, as illustrated in Table 5, and the computer calibration table is updated with this information, as illustrated in Table 6.

10.2.3.3 Routine Calibration - The GC-MS system is calibrated daily (and before sample analysis) with a one-point calibration. The GC-MS system is calibrated either with the dynamic calibration procedure [see Figure 8(a)] or with a 6-L SUMMA<sup>®</sup> passivated canister filled with humid calibration standards from the calibration manifold (see Section 10.2.3.2). After the single point calibration, the GC-MS analytical system is challenged with a humidified zero gas stream to insure the analytical system returns to specification (less than 0.2 ppbv of selective organics).

### 10.3 GC-FID-ECD System Performance Criteria (With Optional PID System) (See Figure 14)

#### 10.3.1 Humid Zero Air Certification

10.3.1.1 Before system calibration and sample analysis, the GC-FID-ECD analytical system is assembled and checked according to manufacturer's instructions.

- 10.3.1.2 The GC-FID-ECD system is first challenged with humid zero air (see Section 12.2.2) and monitored.
- 10.3.1.3 Analytical systems contaminated with less than 0.2 ppbv of targeted VOCs are acceptable.
- 10.3.2 GC Retention Time Windows Determination (See Table 7)
  - 10.3.2.1 Before analysis can be performed, the retention time windows must be established for each analyte.
  - 10.3.2.2 Make sure the GC system is within optimum operating conditions.
  - 10.3.2.3 Make three injections of the standard containing all compounds for retention time window determination. [Note: The retention time window must be established for each analyte every 72 hours during continuous operation.]
  - 10.3.2.4 Calculate the standard deviation of the three absolute retention times for each single component standard. The retention window is defined as the mean plus or minus three times the standard deviation of the individual retention times for each standard. In those cases where the standard deviation for a particular standard is zero, the laboratory must substitute the standard deviation of a closely-eluting, similar compound to develop a valid retention time window.
  - 10.3.2.5 The laboratory must calculate retention time windows for each standard (see Table 7) on each GC column, whenever a new GC column is installed or when major components of the GC are changed. The data must be noted and retained in a notebook by the laboratory as part of the user SOP and as a quality assurance check of the analytical system.

### 10.3.3 GC Calibration

[Note: Initial and routine calibration procedures are illustrated in Figure 14.]

10.3.3.1 Initial Calibration - Initially, a multipoint dynamic calibration (three levels plus humid zero air) is performed on the GC-FID-ECD system, before sample analysis, with the assistance of a calibration system (see Figure 8). The calibration system uses NBS traceable standards or NBS/EPA CRMs in pressurized cylinders [containing a mixture of the targeted VOCs at nominal concentrations of 10 ppmv in nitrogen (Section 8.2)] as working standards to be diluted with humid zero air. The contents of the working standard cylinders are metered ( $2 \text{ cm}^3/\text{min}$ ) into the heated mixing chamber where they are mixed with a 2-L/min humidified zero air stream to achieve a nominal 10 ppbv per compound calibration mixture (see Figure 8). This nominal 10 ppbv standard mixture is allowed to flow and equilibrate for an appropriate amount of time. After the equilibration period, the gas standard mixture is sampled and analyzed by the GC-MS system [see Figure 8(a)]. The results of the analyses are averaged, flow audits are performed on the mass flow controllers used to generate the standards and the appropriate response factors (concentration/area counts) are calculated for each compound, as illustrated in Table 5. [Note: GC-FIDs are linear in the 1-20 ppbv range and may not require repeated multipoint calibrations; whereas, the GC-ECD will require frequent linearity evaluation.] Table 5 outlines typical calibration response factors

and retention times for 40 VOCs. After the GC-FID-ECD is calibrated at the three concentration levels, a second humid zero air sample is passed through the system and analyzed. The second humid zero air test is used to verify that the GC-FID-ECD system is certified clean (less than 0.2 ppbv of target compounds).

10.3.3.2 Routine Calibration - A one point calibration is performed daily on the analytical system to verify the initial multipoint calibration (see Section 10.3.3.1). The analyzers (GC-FID-ECD) are calibrated (before sample analysis) using the static calibration procedures (see Section 10.2.3.2) involving pressurized gas cylinders containing low concentrations of the targeted VOCs (10 ppbv) in nitrogen. After calibration, humid zero air is once again passed through the analytical system to verify residual VOCs are not present.

#### 10.3.4 GC-FID-ECD-PID System Performance Criteria

10.3.4.1 As an option, the user may wish to include a photoionization detector (PID) to assist in peak identification and increase sensitivity.

10.3.4.2 This analytical system is presently being used in U.S. Environmental Protection Agency's Urban Air Toxic Pollutant Program (UATP).

10.3.4.3 Preparation of the GC-FID-ECD-PID analytical system is identical to the GC-FID-ECD system (see Section 10.3).

10.3.4.4 Table 8 outlines typical retention times (minutes) for selected organics using the GC-FID-ECD-PID analytical system.

#### 10.4 Analytical Procedures

##### 10.4.1 Canister Receipt

10.4.1.1 The overall condition of each sample canister is observed. Each canister should be received with an attached sample identification tag.

- 10.4.1.2 Each canister is recorded in the dedicated laboratory logbook. Also noted on the identification tag are date received and initials of recipient.
- 10.4.1.3 The pressure of the canister is checked by attaching a pressure gauge to the canister inlet. The canister valve is opened briefly and the pressure (kPa, psig) is recorded. [Note: If pressure is <83 kPa (<12 psig), the user may wish to pressurize the canisters, as an option, with zero grade nitrogen up to 137 kPa (20 psig) to ensure that enough sample is available for analysis. However, pressurizing the canister can introduce additional error, increase the minimum detection limit (MDL), and is time consuming. The user should weigh these limitations as part of his program objectives before pressurizing.] Final cylinder pressure is recorded on canister sampling field data sheet (see Figure 10).
- 10.4.1.4 If the canister pressure is increased, a dilution factor (DF) is calculated and recorded on the sampling data sheet.

$$DF = \frac{Y_a}{X_a}$$

where:

$X_a$  = canister pressure (kPa, psia) absolute before dilution.

$Y_a$  = canister pressure (kPa, psia) absolute after dilution.

After sample analysis, detected VOC concentrations are multiplied by the dilution factor to determine concentration in the sampled air.

## 10.4.2 GC-MS-SCAN Analysis (With Optional FID System)

- 10.4.2.1 The analytical system should be properly assembled, humid zero air certified (see Section 12.3), operated (see Table 3), and calibrated for accurate VOC determination.
- 10.4.2.2 The mass flow controllers are checked and adjusted to provide correct flow rates for the system.
- 10.4.2.3 The sample canister is connected to the inlet of the GC-MS-SCAN (with optional FID) analytical system. For pressurized samples, a mass flow controller is placed on the canister and the canister valve is opened and the canister flow is vented past a tee inlet to the analytical system at a flow of 75 cm<sup>3</sup>/min so that 40 cm<sup>3</sup>/min is pulled through the Nafion® dryer to the six-port chromatographic valve. [Note: Flow rate is not as important as acquiring sufficient sample volume.] Sub-ambient pressure samples are connected directly to the inlet.
- 10.4.2.4 The GC oven and cryogenic trap (inject position) are cooled to their set points of -50°C and -160°C, respectively.
- 10.4.2.5 As soon as the cryogenic trap reaches its lower set point of -160°C, the six-port chromatographic valve is turned to its fill position to initiate sample collection.
- 10.4.2.6 A ten minute collection period of canister sample is utilized. [Note: 40 cm<sup>3</sup>/min x 10 min = 400 cm<sup>3</sup> sampled canister contents.]
- 10.4.2.7 After the sample is preconcentrated in the cryogenic trap, the GC sampling valve is cycled to the inject position and the cryogenic trap is heated. The trapped analytes are thermally desorbed onto the head of the OV-1 capillary column (0.31 mm I.D. x 50 m length). The GC oven is programmed to start at -50°C and after 2 min to heat to 150°C at a rate of 8°C per minute.

- 10.4.2.8 Upon sample injection onto the column, the MS is signaled by the computer to scan the eluting carrier gas from 18 to 250 amu, resulting in a 1.5 Hz repetition rate. This corresponds to about 6 scans per eluting chromatographic peak.
  - 10.4.2.9 Primary identification is based upon retention time and relative abundance of eluting ions as compared to the spectral library stored on the hard disk of the GC-MS data computer.
  - 10.4.2.10 The concentration (ppbv) is calculated using the previously established response factors (see Section 10.2.3.2), as illustrated in Table 5. [Note: If the canister is diluted before analysis, an appropriate multiplier is applied to correct for the volume dilution of the canister (Section 10.4.1.4).]
  - 10.4.2.11 The optional FID trace allows the analyst to record the progress of the analysis.
- 10.4.3 GC-MS-SIM Analysis (With Optional FID System)
- 10.4.3.1 When the MS is placed in the SIM mode of operation, the MS monitors only preselected ions, rather than scanning all masses continuously between two mass limits.
  - 10.4.3.2 As a result, increased sensitivity and improved quantitative analysis can be achieved.
  - 10.4.3.3 Similar to the GC-MS-SCAN configuration, the GC-MS-SIM analysis is based on a combination of retention times and relative abundances of selected ions (see Table 2 and Table 5). These qualifiers are stored on the hard disk of the GC-MS computer and are applied for identification of each chromatographic peak. Once the GC-MS-SIM has identified the peak, a calibration response factor is used to determine the analyte's concentration.

10.4.3.4 The individual analyses are handled in three phases: data acquisition, data reduction, and data reporting. The data acquisition software is set in the SIM mode, where specific compound fragments are monitored by the MS at specific times in the analytical run. Data reduction is coordinated by the postprocessing macro program that is automatically accessed after data acquisition is completed at the end of the GC run. Resulting ion profiles are extracted, peaks are identified and integrated, and an internal integration report is generated by the program. A reconstructed ion chromatogram for hardcopy reference is prepared by the program and various parameters of interest such as time, date, and integration constants are printed. At the completion of the macro program, the data reporting software is accessed. The appropriate calibration table (see Table 9) is retrieved by the data reporting program from the computer's hard disk storage and the proper retention time and response factor parameters are applied to the macro program's integration file. With reference to certain pre-set acceptance criteria, peaks are automatically identified and quantified and a final summary report is prepared, as illustrated in Table 10.

#### 10.4.4 GC-FID-ECD Analysis (With Optional PID System)

10.4.4.1 The analytical system should be properly assembled, humid zero air certified (see Section 12.2) and calibrated through a dynamic standard calibration procedure (see Section 10.3.2). The FID detector is lit and allowed to stabilize.

10.4.4.2 Sixty-four minutes are required for each sample analysis - 15 min for system initialization, 14 min for sample collection, 30 min for analysis, and 5 min for post-time, during which a report is printed. [Note: This may vary depending

- 10.4.4.3 The helium and sample mass flow controllers are checked and adjusted to provide correct flow rates for the system. Helium is used to purge residual air from the trap at the end of the sampling phase and to carry the revolatilized VOCs from the trap onto the GC column and into the FID-ECD. The hydrogen, burner air, and nitrogen flow rates should also be checked. The cryogenic trap is connected and verified to be operating properly while flowing cryogen through the system.
- 10.4.4.4 The sample canister is connected to the inlet of the GC-FID-ECD analytical system. The canister valve is opened and the canister flow is vented past a tee inlet to the analytical system at 75 cm<sup>3</sup>/min using a 0-500 cm<sup>3</sup>/min Tylan mass flow controller. During analysis, 40 cm<sup>3</sup>/min of sample gas is pulled through the six-port chromatographic valve and routed through the trap at the appropriate time while the extra sample is vented. The VOCs are condensed in the trap while the excess flow is exhausted through an exhaust vent, which assures that the sample air flowing through the trap is at atmospheric pressure.
- 10.4.4.5 The six-port valve is switched to the inject position and the canister valve is closed.
- 10.4.4.6 The electronic integrator is started.
- 10.4.4.7 After the sample is preconcentrated on the trap, the trap is heated and the VOCs are thermally desorbed onto the head of the capillary column. Since the column is at -50°C, the VOCs are cryofocussed on the column. Then, the oven temperature (programmed) increases and the VOCs elute from the column to the parallel FID-ECD assembly.
- 10.4.4.8 The peaks eluting from the detectors are identified by retention time (see Table 7 and Table 8), while peak areas are recorded in area

counts. Figures 15 and 16 illustrate typical response of the FID and ECD, respectively, for the forty (40) targeted VOCs. [Note: Refer to Table 7 for peak number and identification.]

- 10.4.4.9 The response factors (see Section 10.3.3.1) are multiplied by the area counts for each peak to calculate ppbv estimates for the unknown sample. If the canister is diluted before analysis, an appropriate dilution multiplier (DF) is applied to correct for the volume dilution of the canister (see Section 10.4.1.4).
- 10.4.4.10 Depending on the number of canisters to be analyzed, each canister is analyzed twice and the final concentrations for each analyte are the averages of the two analyses.
- 10.4.4.11 However, if the GC-FID-ECD analytical system discovers unexpected peaks which need further identification and attention or overlapping peaks are discovered, eliminating possible quantitation, the sample should then be subjected to a GC-MS-SCAN for positive identification and quantitation.

## 11. Cleaning and Certification Program

### 11.1 Canister Cleaning and Certification

- 11.1.1 All canisters must be clean and free of any contaminants before sample collection.
- 11.1.2 All canisters are leak tested by pressurizing them to approximately 206 kPa (30 psig) with zero air. [Note: The canister cleaning system in Figure 7 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 leak tight, the pressure should not vary more than  $\pm 13.8$  kPa ( $\pm 2$  psig) over the 24 hour period.
- 11.1.3 A canister cleaning system may be assembled as illustrated in Figure 7. 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 (for at least one 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.]

- 11.1.4 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.
- 11.1.5 The zero 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. Steps 11.1.3 through 11.1.5 are repeated two additional times for a total of three (3) evacuation/pressurization cycles for each set of canisters.
- 11.1.6 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 or GC-FID-ECD 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 targets VOCs). The check can then be reduced to a lower percentage of canisters.
- 11.1.7 The canister is reattached to the cleaning manifold and is then reevacuated to  $< 0.05$  mm Hg 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 neck of each canister for field notes and chain-of-custody purposes.

- 11.1.8 As an option to the humid zero air cleaning procedures, the canisters could be heated in an isothermal oven to 100°C during Section 11.1.3 to ensure that lower molecular weight compounds (C<sub>2</sub>-C<sub>8</sub>) are not retained on the walls of the canister. [Note: For sampling heavier, more complex VOC mixtures, the canisters should be heated to 250°C during Section 11.1.3.7.] Once heated, the canisters are evacuated to 0.05 mm Hg. At the end of the heated/evacuated cycle, the canisters are pressurized with humid zero air and analyzed by the GC-FID-ECD system. Any canister that has not tested clean (less than 0.2 ppbv of targeted compounds) should not be used. Once tested clean, the canisters are reevacuated to 0.05 mm Hg and remain in the evacuated state until used.

## 11.2 Sampling System Cleaning and Certification

### 11.2.1 Cleaning Sampling System Components

- 11.2.1.1 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.
- 11.2.1.2 The parts are then rinsed with HPLC grade deionized water and dried in a vacuum oven at 100°C for 12 to 24 hours.
- 11.2.1.3 Once the sampler is assembled, the entire system is purged with humid zero air for 24 hours.

### 11.2.2 Humid 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 of targeted compounds) have occurred when challenged with the test gas stream.]

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

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

11.2.2.3 The humid zero gas stream passes through the calibration manifold, through the sampling system (without an evacuated canister) to a GC-FID-ECD analytical system at 75 cm<sup>3</sup>/min so that 40 cm<sup>3</sup>/min is pulled through the six-port valve and routed through the cryogenic trap (see Section 10.2.2.1) at the appropriate time while the extra sample is vented. [Note: The exit of the sampling system (without the canister) replaces the canister in Figure 4.] After the sample (400 mL) is preconcentrated on the trap, the trap is heated and the VOCs are thermally desorbed onto the head of the capillary column. Since the column is at -50°C, the VOCs are cryofocussed on the column. Then, the oven temperature (programmed) increases and the VOCs begin to elute and are detected by a GC-MS (see Section 10.2) or the GC-FID-ECD (see Section 10.3). The analytical system should not detect greater than 0.2 ppbv of targeted VOCs in order for the sampling system to pass the humid zero air certification

test. Chromatograms of a certified sampler and contaminated sampler are illustrated in Figures 17(a) and (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 11.2.3.

- 11.2.3 Sampler System Certification with Humid Calibration Gas Standards
  - 11.2.3.1 Assemble the dynamic calibration system and manifold as illustrated in Figure 8.
  - 11.2.3.2 Verify that the calibration system is clean (less than 0.2 ppbv of targeted compounds) by sampling a humidified gas stream, without gas calibration standards, with a previously certified clean canister (see Section 12.1).
  - 11.2.3.3 The assembled dynamic calibration system is certified clean if less than 0.2 ppbv of targeted compounds are found.
  - 11.2.3.4 For generating the humidified calibration standards, the calibration gas cylinder(s) (see Section 8.2) containing nominal concentrations of 10 ppmv in nitrogen of selected VOCs, are attached to the calibration system, as outlined in Section 10.2.3.1. The gas cylinders are opened and the gas mixtures are passed through 0 to 10 cm<sup>3</sup>/min certified mass flow controllers to generate ppb levels of calibration standards.
  - 11.2.3.5 After the appropriate equilibrium period, attach the sampling system (containing a certified evacuated canister) to the manifold, as illustrated in Figure 8(a).

- 11.2.3.6 Sample the dynamic calibration gas stream with the sampling system according to Section 9.2.1. [Note: To conserve generated calibration gas, bypass the canister sampling system manifold and attach the sampling system to the calibration gas stream at the inlet of the in-line filter of the sampling system so the flow will be less than 500 cm<sup>3</sup>/min.]
- 11.2.3.7 Concurrent with the sampling system operation, realtime monitoring of the calibration gas stream is accomplished by the on-line GC-MS or GC-multidetector analytical system [Figure 8(b)] to provide reference concentrations of generated VOCs.
- 11.2.3.8 At the end of the sampling period (normally same time period used for anticipated sampling), the sampling system canister is analyzed and compared to the reference GC-MS or GC-multidetector analytical system to determine if the concentration of the targeted VOCs was increased or decreased by the sampling system.
- 11.2.3.9 A recovery of between 90% and 110% is expected for all targeted VOCs.

## 12. Performance Criteria and Quality Assurance

### 12.1 Standard Operating Procedures (SOPs)

- 12.1.1 SOPs should be generated in each laboratory describing and documenting the following activities: (1) assembly, calibration, leak check, and operation of specific sampling systems and equipment used; (2) preparation, storage, shipment, and handling of samples; (3) assembly, leak-check, calibration, and operation of the analytical system, addressing the specific equipment used; (4) canister storage and cleaning; and (5) all aspects of data recording and processing, including lists of computer hardware and software used.

12.1.2 Specific stepwise instructions should be provided in the SOPs and should be readily available to and understood by the laboratory personnel conducting the work.

## 12.2 Method. Relative Accuracy and Linearity

12.2.1 Accuracy can be determined by injecting VOC standards (see Section 8.2) from an audit cylinder into a sampler. The contents are then analyzed for the components contained in the audit canister. Percent relative accuracy is calculated:

$$\% \text{ Relative Accuracy} = \frac{X - Y}{X} \times 100$$

Where: Y = Concentration of the targeted compound recovered from sampler.

X = Concentration of VOC targeted compound in the NBS-SRM or EPA-CRM audit cylinders.

12.2.2 If the relative accuracy does not fall between 90 and 110 percent, the field sampler should be removed from use, cleaned, and recertified according to initial certification procedures outlined in Section 11.2.2 and Section 11.2.3. Historically, concentrations of carbon tetrachloride, tetrachloroethylene, and hexachlorobutadiene have sometimes been detected at lower concentrations when using parallel ECD and FID detectors. When these three compounds are present at concentrations close to calibration levels, both detectors usually agree on the reported concentrations. At concentrations below 4 ppbv, there is a problem with nonlinearity of the ECD. Plots of concentration versus peak area for calibration compounds detected by the ECD have shown that the curves are nonlinear for carbon tetrachloride, tetrachloroethylene, and hexachlorobutadiene, as illustrated in Figures 18(a) through 18(c). Other targeted ECD and FID compounds scaled linearly for the range 0 to 8 ppbv, as shown for chloroform in Figure 18(d). For compounds that are not linear over the calibration

range, area counts generally roll off between 3 and 4 ppbv. To correct for the nonlinearity of these compounds, an additional calibration step is performed. An evacuated stainless steel canister is pressurized with calibration gas at a nominal concentration of 8 ppbv. The sample is then diluted to approximately 3.5 ppbv with zero air and analyzed. The instrument response factor (ppbv/area) of the ECD for each of the three compounds is calculated for the 3.5 ppbv sample. Then, both the 3.5 ppbv and the 8 ppbv response factors are entered into the ECD calibration table. The software for the Hewlett-Packard 5880 level 4 GC is designed to accommodate multilevel calibration entries, so the correct response factors are automatically calculated for concentrations in this range.

## 12.3 Method Modification

### 12.3.1 Sampling

12.3.1.1 The sampling system for pressurized canister sampling could be modified to use a lighter, more compact pump. The pump currently being used weighs about 16 kilograms (35 lbs). Commercially available pumps that could be used as alternatives to the prescribed sampler pump are described below. Metal Bellows MB-41 pump: These pumps are cleaned at the factory; however, some precaution should be taken with the circular (4.8 cm diameter) Teflon® and stainless steel part directly under the flange. It is often dirty when received and should be cleaned before use. This part is cleaned by removing it from the pump, manually cleaning with deionized water, and placing in a vacuum oven at 100°C for at least 12 hours. Exposed parts of the pump head are also cleaned with swabs and allowed to air dry. These pumps have

proven to be very reliable; however, they are only useful up to an outlet pressure of about 137 kPa (20 psig). Neuberger Pump: Viton gaskets or seals must be specified with this pump. The "factory direct" pump is received contaminated and leaky. The pump is cleaned by disassembling the pump head (which consists of three stainless steel parts and two gaskets), cleaning the gaskets with deionized water and drying in a vacuum oven, and remachining (or manually lapping) the sealing surfaces of the stainless steel parts. The stainless steel parts are then cleaned with methanol, hexane, deionized water and heated in a vacuum oven. The cause for most of the problems with this pump has been scratches on the metal parts of the pump head. Once this rework procedure is performed, the pump is considered clean and can be used up to about 240 kPa (35 psig) output pressure. This pump is utilized in the sampling system illustrated in Figure 3.

#### 12.3.1.2 Urban Air Toxics Sampler

The sampling system described in this method can be modified like the sampler in EPA's FY-88 Urban Air Toxics Pollutant Program. This particular sampler is described in Appendix C (see Figure 19).

### 12.3.2 Analysis

12.3.2.1 Inlet tubing from the calibration manifold could be heated to 50°C (same temperature as the calibration manifold) to prevent condensation on the internal walls of the system.

12.3.2.2 The analytical strategy for Method T0-14 involves positive identification and quantitation by GC-MS-SCAN-SIM mode of operation with optional FID. This is a highly specific and sensitive detection technique. Because a specific detector system (GC-MS-SCAN-SIM) is more complicated and expensive than the use of non-specific detectors

(GC-FID-ECD-PID), the analyst may want to perform a screening analysis and preliminary quantitation of VOC species in the sample, including any polar compounds, by utilizing the GC-multidetector (GC-FID-ECD-PID) analytical system prior to GC-MS analysis. This system can be used for approximate quantitation. The GC-FID-ECD-PID provides a "snapshot" of the constituents in the sample, allowing the analyst to determine:

- Extent of misidentification due to overlapping peaks,
- Whether the constituents are within the calibration range of the anticipated GC-MS-SCAN-SIM analysis or does the sample require further dilution, and
- Are there unexpected peaks which need further identification through GC-MS-SCAN or are there peaks of interest needing attention?

If unusual peaks are observed from the GC-FID-ECD-PID system, the analyst then performs a GC-MS-SCAN analysis. The GC-MS-SCAN will provide positive identification of suspect peaks from the GC-FID-ECD-PID system. If no unusual peaks are identified and only a select number of VOCs are of concern, the analyst can then proceed to GC-MS-SIM. The GC-MS-SIM is used for final quantitation of selected VOCs. Polar compounds, however, cannot be identified by the GC-MS-SIM due to the use of a Nafion® dryer to remove water from the sample prior to analysis. The dryer removes polar compounds along with the water. The analyst often has to make this decision incorporating project objectives, detection limits, equipment availability, cost and personnel capability in developing an analytical strategy. Figure 20 outlines the use of the GC-FID-ECD-PID as a "screening" approach, with the GC-MS-SCAN-SIM for final identification and quantitation.

## 12.4 Method Safety

This procedure may involve hazardous materials, operations, and equipment. This method does not purport to address all of the safety problems associated with its use. It is the user's responsibility to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to the implementation of this procedure. This should be part of the user's SOP manual.

## 12.5 Quality Assurance (See Figure 21)

### 12.5.1 Sampling System

- 12.5.1.1 Section 9.2 suggests that a portable GC system be used as a "screening analysis" prior to locating fixed-site samplers (pressurized or subatmospheric).
- 12.5.1.2 Section 9.2 requires pre and post-sampling measurements with a certified mass flow controller for flow verification of sampling system.
- 12.5.1.3 Section 11.1 requires all canisters to be pressure tested to 207 kPa  $\pm$  14 kPa (30 psig  $\pm$  2 psig) over a period of 24 hours.
- 12.5.1.4 Section 11.1 requires that all canisters be certified clean (containing less than 0.2 ppbv of targeted VOCs) through a humid zero air certification program.
- 12.5.1.5 Section 11.2.2 requires all field sampling systems to be certified initially clean (containing less than 0.2 ppbv of targeted VOCs) through a humid zero air certification program.
- 12.5.1.6 Section 11.2.3 requires all field sampling systems to pass an initial humidified calibration gas certification [at VOC concentration levels expected in the field (e.g., 0.5 to 2 ppbv)] with a percent recovery of greater than 90.

### 12.5.2 GC-MS-SCAN-SIM System Performance Criteria

- 12.5.2.1 Section 10.2.1 requires the GC-MS analytical system to be certified clean (less than 0.2

ppbv of targeted VOCs) prior to sample analysis, through a humid zero air certification.

12.5.2.2 Section 10.2.2 requires the daily tuning of the GC-MS with 4-bromofluorobenzene (4-BFB) and that it meet the key ions and ion abundance criteria (10%) outlined in Table 5.

12.5.2.3 Section 10.2.3 requires both an initial multi-point humid static calibration (three levels plus humid zero air) and a daily calibration (one point) of the GC-MS analytical system.

### 12.5.3 GC-Multidetector System Performance Criteria

12.5.3.1 Section 10.3.1 requires the GC-FID-ECD analytical system, prior to analysis, to be certified clean (less than 0.2 ppbv of targeted VOCs) through a humid zero air certification.

12.5.3.2 Section 10.3.2 requires that the GC-FID-ECD analytical system establish retention time windows for each analyte prior to sample analysis, when a new GC column is installed, or major components of the GC system altered since the previous determination.

12.5.3.3 Section 8.2 requires that all calibration gases be traceable to a National Bureau of Standards (NBS) Standard Reference Material (SRM) or to a NBS/EPA approved Certified Reference Material (CRM).

12.5.3.4 Section 10.3.2 requires that the retention time window be established throughout the course of a 72-hr analytical period.

13.5.3.5 Section 10.3.3 requires both an initial multi-point calibration (three levels plus humid zero air) and a daily calibration (one point) of the GC-FID-ECD analytical system with zero gas dilution of NBS traceable or NBS/EPA CRMs gases. [Note: Gas cylinders of VOCs at the ppm and ppb level are available for audits from the USEPA, Environmental Monitoring Systems

Laboratory, Quality Assurance Division, MD-77B,  
Research Triangle Park, NC 27711, (919)541-4531.

Appendix A outlines five groups of audit gas  
cylinders available from USEPA.]

13. Acknowledgements

The determination of volatile and some semi-volatile organic compounds in ambient air is a complex task, primarily because of the wide variety of compounds of interest and the lack of standardized sampling and analytical procedures. While there are numerous procedures for sampling and analyzing VOCs/SVOCs in ambient air, this method draws upon the best aspects of each one and combines them into a standardized methodology. To that end, the following individuals contributed to the research, documentation and peer review of this manuscript.

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T014-56

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T014-57

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TABLE 1. VOLATILE ORGANIC COMPOUND DATA SHEET

COMPOUND (SYNONYM)	FORMULA	MOLECULAR WEIGHT	BOILING POINT (°C)	MELTING POINT (°C)	CAS NUMBER
Freon 12 (Dichlorodifluoromethane)	$\text{Cl}_2\text{CF}_2$	120.91	-29.8	-158.0	
Methyl chloride (Chloromethane)	$\text{CH}_3\text{Cl}$	50.49	-24.2	-97.1	74-87-3
Freon 114 (1,2-Dichloro-1,1,2,2-tetrafluoroethane)	$\text{ClCF}_2\text{CClF}_2$	170.93	4.1	-94.0	
Vinyl chloride (Chloroethylene)	$\text{CH}_2=\text{CHCl}$	62.50	-13.4	-1538.0	75-01-4
Methyl bromide (Bromomethane)	$\text{CH}_3\text{Br}$	94.94	3.6	-93.6	74-83-9
Ethyl chloride (Chloroethane)	$\text{CH}_3\text{CH}_2\text{Cl}$	64.52	12.3	-136.4	75-00-3
Freon 11 (Trichlorofluoromethane)	$\text{CCl}_3\text{F}$	137.38	23.7	-111.0	
Vinylidene chloride (1,1-Dichloroethene)	$\text{C}_2\text{H}_2\text{Cl}_2$	96.95	31.7	-122.5	75-35-4
Dichloromethane (Methylene chloride)	$\text{CH}_2\text{Cl}_2$	84.94	39.8	-95.1	75-09-2
Freon 113 (1,1,2-Trichloro-1,2,2-trifluoroethane)	$\text{CF}_2\text{ClCCl}_2\text{F}$	187.38	47.7	-36.4	
1,1-Dichloroethane (Ethylidene chloride)	$\text{CH}_3\text{CHCl}_2$	98.96	57.3	-97.0	74-34-3
cis-1,2-Dichloroethylene	$\text{CHCl}=\text{CHCl}$	96.94	60.3	-80.5	
Chloroform (Trichloromethane)	$\text{CHCl}_3$	119.38	61.7	-63.5	67-66-3
1,2-Dichloroethane (Ethylene dichloride)	$\text{ClCH}_2\text{CH}_2\text{Cl}$	98.96	83.5	-35.3	107-06-2
Methyl chloroform (1,1,1-Trichloroethane)	$\text{CH}_3\text{CCl}_3$	133.41	74.1	-30.4	71-55-6
Benzene (Cyclohexatriene)	$\text{C}_6\text{H}_6$	78.12	80.1	5.5	71-43-2
Carbon tetrachloride (Tetrachloromethane)	$\text{CCl}_4$	153.82	76.5	-23.0	56-23-5
1,2-Dichloropropane (Propylene dichloride)	$\text{CH}_3\text{CHClCH}_2\text{Cl}$	112.99	96.4	-100.4	78-87-5
Trichloroethylene (Trichloroethene)	$\text{ClCH}=\text{CCl}_2$	131.29	87	-73.0	79-01-6
cis-1,3-Dichloropropene (cis-1,3-dichloropropylene)	$\text{CH}_3\text{CCl}=\text{CHCl}$	110.97	76		

T014-60

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

COMPOUND (SYNONYM)	FORMULA	MOLECULAR WEIGHT	BOILING POINT(°C)	MELTING POINT (°C)	CAS NUMBER
trans-1,3-Dichloropropene (cis-1,3-Dichloropropylene)	$\text{ClCH}_2\text{CH}=\text{CHCl}$	110.97	112.0		
1,1,2-Trichloroethane (Vinyl trichloride)	$\text{CH}_2\text{ClCHCl}_2$	133.41	113.8	-36.5	79-00-5
Toluene (Methyl benzene)	$\text{C}_6\text{H}_5\text{CH}_3$	92.15	110.6	-95.0	108-88-3
1,2-Dibromoethane (Ethylene dibromide)	$\text{BrCH}_2\text{CH}_2\text{Br}$	187.88	131.3	9.8	106-93-4
Tetrachloroethylene (Perchloroethylene)	$\text{Cl}_2\text{C}=\text{CCl}_2$	165.83	121.1	-19.0	127-18-4
Chlorobenzene (Phenyl chloride)	$\text{C}_6\text{H}_5\text{Cl}$	112.56	132.0	-45.6	108-90-7
Ethylbenzene	$\text{C}_6\text{H}_5\text{C}_2\text{H}_5$	106.17	136.2	-95.0	100-41-4
m-Xylene (1,3-Dimethylbenzene)	$1,3-(\text{CH}_3)_2\text{C}_6\text{H}_4$	106.17	139.1	-47.9	
p-Xylene (1,4-Dimethylxylene)	$1,4-(\text{CH}_3)_2\text{C}_6\text{H}_4$	106.17	138.3	13.3	
Styrene (Vinyl benzene)	$\text{C}_6\text{H}_5\text{CH}=\text{CH}_2$	104.16	145.2	-30.6	100-42-5
1,1,2,2-Tetrachloroethane	$\text{CHCl}_2\text{CHCl}_2$	167.85	146.2	-36.0	79-34-5
o-Xylene (1,2-Dimethylbenzene)	$1,2-(\text{CH}_3)_2\text{C}_6\text{H}_4$	106.17	144.4	-25.2	
1,3,5-Trimethylbenzene (Mesitylene)	$1,3,5-(\text{CH}_3)_3\text{C}_6\text{H}_6$	120.20	164.7	-44.7	108-67-8
1,2,4-Trimethylbenzene (Pseudocumene)	$1,2,4-(\text{CH}_3)_3\text{C}_6\text{H}_6$	120.20	169.3	-43.8	95-63-6
m-Dichlorobenzene (1,3-Dichlorobenzene)	$1,3-\text{Cl}_2\text{C}_6\text{H}_4$	147.01	173.0	-24.7	541-73-1
Benzyl chloride ( $\alpha$ -Chlorotoluene)	$\text{C}_6\text{H}_5\text{CH}_2\text{Cl}$	126.59	179.3	-39.0	100-44-7
o-Dichlorobenzene (1,2-Dichlorobenzene)	$1,2-\text{Cl}_2\text{C}_6\text{H}_4$	147.01	180.5	-17.0	95-50-1
p-Dichlorobenzene (1,4-Dichlorobenzene)	$1,4-\text{Cl}_2\text{C}_6\text{H}_4$	147.01	174.0	53.1	106-46-7
1,2,4-Trichlorobenzene	$1,2,4-\text{Cl}_3\text{C}_6\text{H}_3$	181.45	213.5	17.0	120-82-1
Hexachlorobutadiene (1,1,2,3,4,4-Hexachloro-1,3-butadiene)					

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

Compound	Ion/Abundance (amu/% base peak)	Expected Retention Time (min)
Freon 12 (Dichlorodifluoromethane)	85/100	5.01
	87/ 31	
Methyl chloride (Chloromethane)	50/100	5.69
	52/ 34	
Freon 114 (1,2-Dichloro-1,1,2,2-tetrafluoroethane)	85/100	6.55
	135/ 56	
	87/ 33	
Vinyl chloride (Chloroethene)	62/100	6.71
	27/125	
	64/ 32	
Methyl bromide (Bromomethane)	94/100	7.83
	96/ 85	
Ethyl chloride (Chloroethane)	64/100	8.43
	29/140	
	27/140	
Freon 11 (Trichlorofluoromethane)	101/100	9.97
	103/ 67	
Vinylidene chloride (1,1-Dichloroethylene)	61/100	10.93
	96/ 55	
	63/ 31	
Dichloromethane (Methylene chloride)	49/100	11.21
	84/ 65	
	86/ 45	
Freon 113 (1,1,2-Trichloro-1,2,2-trifluoroethane)	151/100	11.60
	101/140	
	103/ 90	
1,1-Dichloroethane (Ethylidene dichloride)	63/100	12.50
	27/ 64	
	65/ 33	
cis-1,2-Dichloroethylene	61/100	13.40
	96/ 60	
	98/ 44	
Chloroform (Trichloromethane)	83/100	13.75
	85/ 65	
	47/ 35	
1,2-Dichloroethane (Ethylene dichloride)	62/100	14.39
	27/ 70	
	64/ 31	
Methyl chloroform (1,1,1-Trichloroethane)	97/100	14.62
	99/ 64	
	61/ 61	
Benzene (Cyclohexatriene)	78/100	15.04
	77/ 25	
	50/ 35	
Carbon tetrachloride (Tetrachloromethane)	117/100	15.18
	119/ 97	

(continued)

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

Compound	Ion/Abundance (amu/% base peak)	Estimated Retention Time (min)
1,2-Dichloropropane (Propylene dichloride)	63/100 41/ 90 62/ 70	15.83
Trichloroethylene (Trichloroethene)	130/100 132/ 92 95/ 87	16.10
cis-1,3-Dichloropropene	75/100 39/ 70 77/ 30	16.96
trans-1,3-Dichloropropene (1,3 dichloro-1-propene)	75/100 39/ 70 77/ 30	17.49
1,1,2-Trichloroethane (Vinyl trichloride)	97/100 83/ 90 61/ 82	17.61
Toluene (Methyl benzene)	91/100 92/ 57	17.86
1,2-Dibromoethane (Ethylene dibromide)	107/100 109/ 96 27/115	18.48
Tetrachloroethylene (Perchloroethylene)	166/100 164/ 74 131/ 60	19.01
Chlorobenzene (Benzene chloride)	112/100 77/ 62 114/ 32	19.73
Ethylbenzene	91/100 106/ 28	20.20
m,p-Xylene(1,3/1,4-dimethylbenzene)	91/100 106/ 40	20.41
Styrene (Vinyl benzene)	104/100 78/ 60 103/ 49	20.81
1,1,2,2-Tetrachloroethane (Tetrachloroethane)	83/100 85/ 64	20.92
o-Xylene (1,2-Dimethylbenzene)	91/100 106/ 40	20.92
4-Ethyltoluene	105/100 120/ 29	22.53
1,3,5-Trimethylbenzene (Mesitylene)	105/100 120/ 42	22.65
1,2,4-Trimethylbenzene (Pseudocumene)	105/100 120/ 42	23.18
m-Dichlorobenzene (1,3-Dichlorobenzene)	146/100 148/ 65 111/ 40	23.31

(continued)

T014-64

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

Compound	Ion/Abundance (amu/% base peak)	Expected Retention Time (min)
Benzyl chloride ( $\alpha$ -Chlorotoluene)	91/100	23.32
p-Dichlorobenzene (1,4-Dichlorobenzene)	126/ 26	23.41
	146/100	
	148/ 65	
o-Dichlorobenzene (1,2-Dichlorobenzene)	111/ 40	23.88
	146/100	
	148/ 65	
1,2,4-Trichlorobenzene	111/ 40	26.71
	180/100	
	182/ 98	
Hexachlorobutadiene (1,1,2,3,4,4 Hexachloro-1,3-butadiene)	184/ 30	27.68
	225/100	
	227/ 66	
	223/ 60	

TABLE 3. GENERAL GC AND MS OPERATING CONDITIONS

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Chromatography

Column	Hewlett-Packard OV-1 crosslinked methyl silicone (50 m x 0.31-mm I.D., 17 $\mu$ m film thickness), or equivalent
Carrier Gas	Helium (2.0 $\text{cm}^3/\text{min}$ at 250°C)
Injection Volume	Constant (1-3 $\mu\text{L}$ )
Injection Mode	Splitless

Temperature Program

Initial Column Temperature	-50°C
Initial Hold Time	2 min
Program	8°C/min to 150°C
Final Hold Time	15 min

Mass Spectrometer

Mass Range	18 to 250 amu
Scan Time	1 sec/scan
EI Condition	70 eV
Mass Scan	Follow manufacturer's instruction for selecting mass selective detector (MS) and selected ion monitoring (SIM) mode
Detector Mode	Multiple ion detection

FID System (Optional)

Hydrogen Flow	30 $\text{cm}^3/\text{minute}$
Carrier Flow	30 $\text{cm}^3/\text{minute}$
Burner Air	400 $\text{cm}^3/\text{minute}$

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

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

TABLE 5. RESPONSE FACTORS (ppbv/area count) AND  
EXPECTED RETENTION TIME FOR GC-MS-SIM  
ANALYTICAL CONFIGURATION

Compounds	Response Factor (ppbv/area count)	Expected Retention Time (minutes)
Freon 12	0.6705	5.01
Methyl chloride	4.093	5.64
Freon 114	0.4928	6.55
Vinyl chloride	2.343	6.71
Methyl bromide	2.647	7.83
Ethyl chloride	2.954	8.43
Freon 11	0.5145	9.87
Vinylidene chloride	1.037	10.93
Dichloromethane	2.255	11.21
Trichlorotrifluoroethane	0.9031	11.60
1,1-Dichloroethane	1.273	12.50
cis-1,2-Dichloroethylene	1.363	13.40
Chloroform	0.7911	13.75
1,2-Dichloroethane	1.017	14.39
Methyl chloroform	0.7078	14.62
Benzene	1.236	15.04
Carbon tetrachloride	0.5880	15.18
1,2-Dichloropropane	2.400	15.83
Trichloroethylene	1.383	16.10
cis-1,3-Dichloropropene	1.877	16.96
trans-1,3-Dichloropropene	1.338	17.49
1,1,2-Trichloroethane	1.891	17.61
Toluene	0.9406	17.86
1,2-Dibromoethane (EDB)	0.8662	18.48
Tetrachloroethylene	0.7357	19.01
Chlorobenzene	0.8558	19.73
Ethylbenzene	0.6243	20.20
m,p-Xylene	0.7367	20.41
Styrene	1.888	20.80
1,1,2,2-Tetrachloroethane	1.035	20.92
o-Xylene	0.7498	20.92
4-Ethyltoluene	0.6181	22.53
1,3,5-Trimethylbenzene	0.7088	22.65
1,2,4-Trimethylbenzene	0.7536	23.18
m-Dichlorobenzene	0.9643	23.31
Benzyl chloride	1.420	23.32
p-Dichlorobenzene	0.8912	23.41
o-Dichlorobenzene	1.004	23.88
1,2,4-Trichlorobenzene	2.150	26.71
Hexachlorobutadiene	0.4117	27.68

TABLE 6. GC-MS-SIM CALIBRATION TABLE

\*\*\* External Standard \*\*\*

Operator: JDF

8 Jan 87 10:02 am

Sample Info : SYR 1

Misc Info:

Integration File Name : DATA:SYR2A02A.I

Sequence Index: 1

Bottle Number : 2

Last Update: 8 Jan 87 8:13 am

Reference Peak Window: 5.00 Absolute Minutes

Non-Reference Peak Window: 0.40 Absolute Minutes

Sample Amount: 0.000 Uncalibrated Peak RF: 0.000 Multiplier: 1.667

Peak Num	Int Type	Ret Time	Signal Description	Compound Name	Area	Amount
1	1 PP	5.020	Mass 85.00 amu	FREON 12	12893	4011 pptv
2	1 PP	5.654	Mass 50.00 amu	METHYLCHLORI	4445	2586 pptv
3	1 BF	6.525	Mass 85.00 amu	FREON 114	7067	1215 pptv
4	1 PB	6.650	Mass 62.00 amu	VINYLCHLORID	2892	1929 pptv
5	1 BF	7.818	Mass 94.00 amu	METHYLBROMID	2401	1729 pptv
6	1 BB	8.421	Mass 64.00 amu	ETHYLCHLORID	2134	2769 pptv
7	1 BV	9.940	Mass 101.00 amu	FREON 11	25069	6460 pptv
8	1 BF	10.869	Mass 61.00 amu	VINDENECHLOR	5034	1700 pptv
9	1 BF	11.187	Mass 49.00 amu	DICHLOROMETH	4803	2348 pptv
10	1 PP	11.225	Mass 41.00 amu	ALLYLCHLORID	761	8247 pptv
11	1 BF	11.578	Mass 151.00 amu	3CHL3FLUETHA	5477	1672 pptv
12	1 BF	12.492	Mass 63.00 amu	1,1DICHLOETH	5052	1738 pptv
13	1 VP	13.394	Mass 61.00 amu	c-1,2DICHLET	4761	1970 pptv
14	1 PH	13.713	Mass 83.00 amu	CHLOROFORM	5327	1678 pptv
15	1 BF	14.378	Mass 62.00 amu	1,2DICHLETHA	5009	2263 pptv
16	1 PB	14.594	Mass 97.00 amu	METHCHLOROFO	6656	2334 pptv
17	1 VP	15.009	Mass 78.00 amu	BENZENE	8352	2167 pptv
18	1 VP	15.154	Mass 117.00 amu	CARBONTETRAC	5888	1915 pptv
19	1 BB	15.821	Mass 63.00 amu	1,2DICHLPROP	3283	1799 pptv
20	1 BB	16.067	Mass 130.00 amu	TRICHLETHENE	4386	2109 pptv
21	1 PB	16.941	Mass 75.00 amu	c-1,3DICHLPR	2228	987.3 pptv
22	1 BF	17.475	Mass 75.00 amu	t-1,3DICHLPR	1626	689.2 pptv
23	1 BB	17.594	Mass 97.00 amu	1,1,2CHLETHA	2721	1772 pptv
24	1 BV	17.844	Mass 91.00 amu	TOLUENE	14417	2733 pptv
25	1 PB	18.463	Mass 107.00 amu	EDB	4070	1365 pptv
26	1 PH	18.989	Mass 166.00 amu	TETRACHLETHE	6874	2065 pptv
27	1 PB	19.705	Mass 112.00 amu	CHLOROBENZEN	5648	1524 pptv
28	1 BF	20.168	Mass 91.00 amu	ETHYLBENZENE	11084	1842 pptv
29	1 PB	20.372	Mass 91.00 amu	m,p-XYLENE	17989	3790 pptv
30	1 BV	20.778	Mass 104.00 amu	STYRENE	3145	1695 pptv
31	1 BH	20.887	Mass 80.00 amu	TETRACHLETHA	4531	1376 pptv
32	1 BF	20.892	Mass 91.00 amu	o-XYLENE	9798	2010 pptv
33	1 VV	22.488	Mass 105.00 amu	4-ETHYLTOLUE	7694	1481 pptv
34	1 VB	22.609	Mass 105.00 amu	1,3,5METHBEN	6781	1705 pptv
35	1 BB	23.144	Mass 95.00 amu	1,2,4METHBEN	7892	2095 pptv
36	1 BV	23.273	Mass 86.00 amu	m-DICHLBENZE	3046	1119 pptv
37	1 VV	23.279	Mass 91.00 amu	BENZYLCHLORI	3880	1006 pptv
38	1 VB	23.378	Mass 86.00 amu	p-DICHLBENZE	6090	2164 pptv
39	1 BF	23.850	Mass 86.00 amu	o-DICHLBENZE	2896	1249 pptv
40	1 BB	26.673	Mass 180.00 amu	1,2,4CHLBENZ	562	767.1 pptv
41	1 BB	27.637	Mass 225.00 amu	HEXACHLBUTAD	6309	1789 pptv

TABLE 7. TYPICAL RETENTION TIME (MIN) AND CALIBRATION RESPONSE FACTORS (ppbv/area count) FOR TARGETED VOCs ASSOCIATED WITH FID AND ECD ANALYTICAL SYSTEM

Peak Number <sup>1</sup>	Compound	Retention Time (RT), minutes	FID	ECD
			Response Factor (RF) (ppbv/area count)	Response Factor (ppbv/area count x 10 <sup>-5</sup> )
1	Freon 12	3.65	3.465	13.89
2	Methyl chloride	4.30	0.693	
3	Freon 114	5.13	0.578	22.32
4	Vinyl chloride	5.28	0.406	
5	Methyl bromide	6.44		26.34
6	Ethyl chloride	7.06	0.413	
7	Freon 11	8.60	6.367	1.367
8	Vinylidene chloride	9.51	0.347	
9	Dichloromethane	9.84	0.903	
10	Trichlorotrifluoroethane	10.22	0.374	3.955
11	1,1-Dichloroethane	11.10	0.359	
12	cis-1,2-Dichloroethylene	11.99	0.368	
13	Chloroform	12.30	1.059	11.14
14	1,2-Dichloroethane	12.92	0.409	
15	Methyl chloroform	13.12	0.325	3.258
16	Benzene	13.51	0.117	
17	Carbon tetrachloride	13.64	1.451	1.077
18	1,2-Dichloropropane	14.26	0.214	
19	Trichloroethylene	14.50	0.327	8.910
20	cis-1,3-Dichloropropene	15.31		
21	trans-1,3-Dichloropropene	15.83		
22	1,1,2-Trichloroethane	15.93	0.336	
23	Toluene	16.17	0.092	
24	1,2-Dibromoethane (EDB)	16.78	0.366	5.137
25	Tetrachloroethylene	17.31	0.324	1.449
26	Chlorobenzene	18.03	0.120	
27	Ethylbenzene	18.51	0.092	
28	m,p-Xylene	18.72	0.095	
29	Styrene	19.12	0.143	
30	1,1,2,2-Tetrachloroethane	19.20		9.856
31	o-Xylene	19.23		
32	4-Ethyltoluene	20.82	0.100	
33	1,3,5-Trimethylbenzene	20.94	0.109	
34	1,2,4-Trimethylbenzene	21.46	0.111	
35	m-Dichlorobenzene	21.50		
36	Benzyl chloride	21.56		
37	p-Dichlorobenzene	21.67	0.188	
38	o-Dichlorobenzene	22.12	0.188	
39	1,2,4-Trichlorobenzene	24.88	0.667	
40	Hexachlorobutadiene	25.82	0.305	1.055

<sup>1</sup> Refer to Figures 15 and 16 for peak location

TABLE 8. TYPICAL RETENTION TIME (minutes) FOR  
SELECTED ORGANICS USING GC-FID-ECD-PID\*  
ANALYTICAL SYSTEM

Compound	Retention Time (minutes)		
	FID	ECD	PID
Acetylene	2.984	--	--
1,3-Butadiene	3.599	--	3.594
Vinyl chloride	3.790	--	3.781
Chloromethane	5.137	--	--
Chloroethane	5.738	--	--
Bromoethane	8.154	--	--
Methylene Chloride	9.232	--	9.218
trans-1,2-Dichloroethylene	10.077	--	10.065
1,1-Dichloroethane	11.190	--	--
Chloroprene	11.502	--	11.491
Perfluorobenzene	13.077	13.078	13.069
Bromochloromethane	13.397	13.396	13.403
Chloroform	13.768	13.767	13.771
1,1,1-Trichloroethane	14.151	14.153	14.158
Carbon Tetrachloride	14.642	14.667	14.686
Benzene/1,2-Dichloroethane	15.128	--	15.114
Perfluorotoluene	15.420	15.425	15.412
Trichloroethylene	17.022	17.024	17.014
1,2-Dichloropropene	17.491	17.805	17.522
Bromodichloromethane	18.369	--	--
trans-1,3-Dichloropropylene	19.694	19.693	19.688
Toluene	20.658	--	20.653
cis-1,3-Dichloropropylene	21.461	21.357	21.357
1,1,2-Trichloroethane	21.823	--	--
Tetrachloroethylene	22.340	22.346	22.335
Dibromochloromethane	22.955	22.959	22.952
Chlorobenzene	24.866	--	24.861
m/p-Xylene	25.763	--	25.757
Styrene/o-Xylene	27.036	--	27.030
Bromofluorobenzene	28.665	28.663	28.660
1,1,2,2-Tetrachloroethane	29.225	29.227	29.228
m-Dichlorobenzene	32.347	32.345	32.342
p-Dichlorobenzene	32.671	32.669	32.666
o-Dichlorobenzene	33.885	33.883	33.880

\* Varian® 3700 GC equipped with J & W Megabore® DB 624 Capillary Column (30 m X 0.53 I.D. mm) using helium carrier gas.

TABLE 9. GC-MS-SIM CALIBRATION TABLE

Last Update: 18 Dec 86 7:54 am  
 Reference Peak Window: 5.00 Absolute Minutes  
 Non-Reference Peak Window: 0.40 Absolute Minutes  
 Sample Amount: 0.000 Uncalibrated Peak RF: 0.000 Multiplier: 1.000

Ret Time	Pk#	Signal	Descr	Amt	pptv	Lvl	[Area]	Pk-Type	Partial Name
5.008	1	Mass	85.00 amu	13620		1	72974	1	FREON 12
5.690	2	Mass	50.00 amu	12720		1	36447	1	METHYLCHLORID
6.552	3	Mass	85.00 amu	8360		1	81251	1	FREON 114
6.709	4	Mass	62.00 amu	8050		1	20118	1	VINYLCHLORIDE
7.831	5	Mass	94.00 amu	12210		1	28265	1	METHYLBROMIDE
8.431	6	Mass	64.00 amu	12574		1	16149	1	ETHYLCHLORIDE
9.970	7	Mass	101.00 amu	12380		1	80088	1	FREON 11
10.927	8	Mass	61.00 amu	7890		1	38954	1	VINDENECHLORI
11.209	9	Mass	49.00 amu	12760		1	43507	1	DICHLOROMETHA
11.331	10	Mass	41.00 amu	12650		1	1945	1	ALLYLCHLORIDE
11.595	11	Mass	151.00 amu	7420		1	40530	1	3CHL3FLUETHAN
12.502	12	Mass	63.00 amu	12710		1	61595	1	1,1DICHLOETHA
13.403	13	Mass	61.00 amu	12630		1	50900	1	c-1,2DICHLETH
13.747	14	Mass	83.00 amu	7670		1	40585	1	CHLOROFORM
14.387	15	Mass	62.00 amu	9040		1	33356	1	1,2DICHLETHAN
14.623	16	Mass	97.00 amu	8100		1	38503	1	METHCHLOROFOR
15.038	17	Mass	78.00 amu	10760		1	69119	1	BENZENE
15.183	18	Mass	117.00 amu	8340		1	42737	1	CARBONTETRACH
15.829	19	Mass	63.00 amu	12780		1	38875	1	1,2DICHLPROPA
16.096	20	Mass	130.00 amu	8750		1	30331	1	TRICHLETHENE
16.956	21	Mass	75.00 amu	4540		1	17078	1	c-1,3DICHLPRO
17.492	22	Mass	75.00 amu	3380		1	13294	1	t-1,3DICHLPRO
17.610	23	Mass	97.00 amu	12690		1	32480	1	1,1,2CHLETHAN
17.862	24	Mass	91.00 amu	10010		1	88036	1	TOLUENE
18.485	25	Mass	107.00 amu	6710		1	33350	1	EDB
19.012	26	Mass	166.00 amu	7830		1	43454	1	TETRACHLETHEN
19.729	27	Mass	112.00 amu	7160		1	44224	1	CHLOROENZENE
20.195	28	Mass	91.00 amu	12740		1	127767	1	ETHYLBENZENE
20.407	29	Mass	91.00 amu	25400		1	200973	1	m,p-XYLENE
20.806	30	Mass	104.00 amu	12390		1	38332	1	STYRENE
20.916	31	Mass	83.00 amu	11690		1	64162	1	TETRACHLETHAN
20.921	32	Mass	91.00 amu	11085		1	90096	1	o-XYLENE
22.528	33	Mass	105.00 amu	12560		1	108747	1	4-ETHYLTOLUEN
22.648	34	Mass	105.00 amu	12620		1	83666	1	1,3,5METHBENZ
23.179	35	Mass	105.00 amu	12710		1	79833	1	1,2,4METHBENZ
23.307	36	Mass	146.00 amu	12650		1	57409	1	m-DICHLBENZEN
23.317	37	Mass	91.00 amu	7900		1	50774	1	BENZYLCHLORID
23.413	38	Mass	146.00 amu	12390		1	58127	1	p-DICHLBENZEN
23.885	39	Mass	146.00 amu	13510		1	52233	1	o-DICHLBENZEN
26.714	40	Mass	180.00 amu	15520		1	18967	1	1,2,4CHLLENZE
27.680	41	Mass	225.00 amu	7470		1	43920	1	HEXACHLBTADI

T014-72

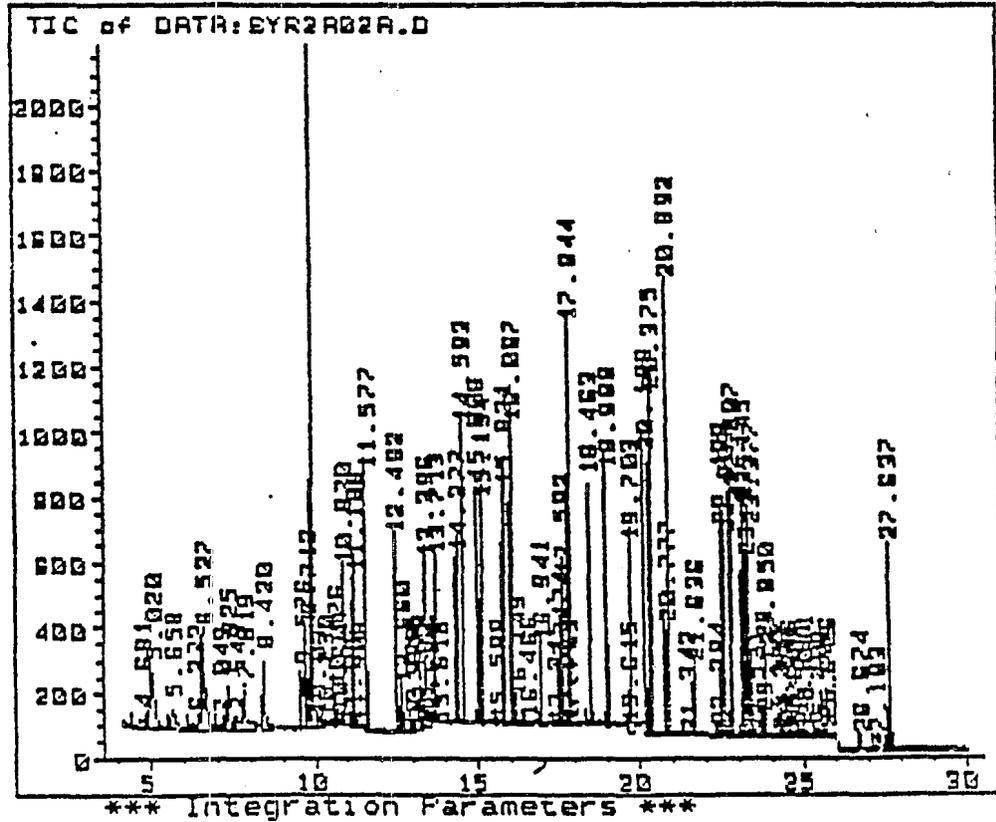
TABLE 10. EXAMPLE OF HARD-COPY OF GC-MS-SIM ANALYSIS

Data file: DATA:SYR2A02A.D  
File type: GC / MS DATA FILE

Name Info: SYR 1  
Misc Info:  
Operator : JDF

Date : 8 Jan 87 10:02 am  
Instrument: MS\_5970  
Inlet : GC

Sequence index : 1  
Alc bottle num : 2  
Replicate num : 1



FALSE : Shoulder Detection Enabled  
0.020 : Expected Peak Width (Min)  
11 : Initial Peak Detection Threshold

4.000	THRESHOLD	5.000
4.000	PEAK_WIDTH	0.200
9.800	PEAK_WIDTH	0.060

TABLE 10. EXAMPLE OF HARD-COPY OF GC-MS-SIM ANALYSIS (cont.)

Operator: JDP

8 Jan 87 10:02 am

Sample Info: SYR 1

Misc Info:

Integration File Name: DATA:SYR2A02A.I

Sequence Index: 1

Bottle Number: 2

Last Update: 8 Jan 87 8:13 am

Reference Peak Window: 5.00 Absolute Minutes

Non-Reference Peak Window: 0.40 Absolute Minutes

Sample Amount: 0.000 Uncalibrated Peak RF: 0.000 Multiplier: 1.667

Peak Num	Int Type	Ret Time	Signal Description	Compound Name	Area	Amount
1	1 PP	5.020	Mass 85.00 amu	FREON 12	12893	4011 pptv
2	1 PP	5.654	Mass 50.00 amu	METHYLCHLORI	4445	2586 pptv
3	1 BP	6.525	Mass 85.00 amu	FREON 114	7067	1215 pptv
4	1 PB	6.650	Mass 62.00 amu	VINYLCHLORID	2892	1929 pptv
5	1 BP	7.818	Mass 94.00 amu	METHYLBROMID	2401	1729 pptv
6	1 BB	8.421	Mass 64.00 amu	ETHYLCHLORID	2134	2769 pptv
7	1 BV	9.940	Mass 101.00 amu	FREON 11	25069	6460 pptv
8	1 BP	10.869	Mass 61.00 amu	VINDENECHLOR	5034	1700 pptv
9	1 BP	11.187	Mass 49.00 amu	DICHLOROMETH	4803	2348 pptv
10	1 PP	11.225	Mass 41.00 amu	ALLYLCHLORID	761	8247 pptv
11	1 BP	11.578	Mass 151.00 amu	3CHL3FLUETHA	5477	1672 pptv
12	1 BP	12.492	Mass 63.00 amu	1,1DICHLOETH	5052	1738 pptv
13	1 VP	13.394	Mass 61.00 amu	c-1,2DICHLET	4761	1970 pptv
14	1 PH	13.713	Mass 83.00 amu	CHLOROFORM	5327	1678 pptv
15	1 BP	14.378	Mass 62.00 amu	1,2DICHLETHA	5009	2263 pptv
16	1 PB	14.594	Mass 97.00 amu	METHCHLOROFO	6656	2334 pptv
17	1 VP	15.009	Mass 78.00 amu	BENZENE	8352	2167 pptv
18	1 VP	15.154	Mass 117.00 amu	CARBONTETRAC	5888	1915 pptv
19	1 BB	15.821	Mass 63.00 amu	1,2DICHLPROP	3283	1799 pptv
20	1 BB	16.067	Mass 130.00 amu	TRICHLETHENE	4386	2109 pptv
21	1 PB	16.941	Mass 75.00 amu	c-1,3DICHLPR	2228	987.3 pptv
22	1 BP	17.475	Mass 75.00 amu	t-1,3DICHLPR	1626	689.2 pptv
23	1 BB	17.594	Mass 97.00 amu	1,1,2CHLETHA	2721	1772 pptv
24	1 BV	17.844	Mass 91.00 amu	TOLUENE	14417	2733 pptv
25	1 PB	18.463	Mass 107.00 amu	EDB	4070	1365 pptv
26	1 PH	18.989	Mass 166.00 amu	TETRACHLETHE	6674	2065 pptv
27	1 PB	19.705	Mass 112.00 amu	CHLOROBENZEN	5648	1524 pptv
28	1 BP	20.168	Mass 91.00 amu	ETHYLBENZENE	11084	1842 pptv
29	1 PB	20.372	Mass 91.00 amu	m,p-XYLENE	17989	3790 pptv
30	1 BV	20.778	Mass 104.00 amu	STYRENE	3145	1695 pptv
31	1 BH	20.887	Mass 83.00 amu	TETRACHLETHA	4531	1376 pptv
32	1 BP	20.892	Mass 91.00 amu	o-XYLENE	9798	2010 pptv
33	1 VV	22.488	Mass 105.00 amu	4-ETHYLTOLUE	7694	1481 pptv
34	1 VB	22.609	Mass 105.00 amu	1,3,5METHBEN	6781	1705 pptv
35	1 BB	23.144	Mass 105.00 amu	1,2,4METHBEN	7892	2095 pptv
36	1 BV	23.273	Mass 146.00 amu	m-DICHLBENZE	3046	1119 pptv
37	1 VV	23.279	Mass 91.00 amu	BENZYLCHLORI	3880	1006 pptv
38	1 VB	23.378	Mass 146.00 amu	p-DICHLBENZE	6090	2164 pptv
39	1 BP	23.850	Mass 146.00 amu	o-DICHLBENZE	2896	1249 pptv
40	1 BB	26.673	Mass 180.00 amu	1,2,4CHLBENZ	562	767.1 pptv
41	1 BB	27.637	Mass 225.00 amu	HEXACHLBUTAD	6309	1789 pptv

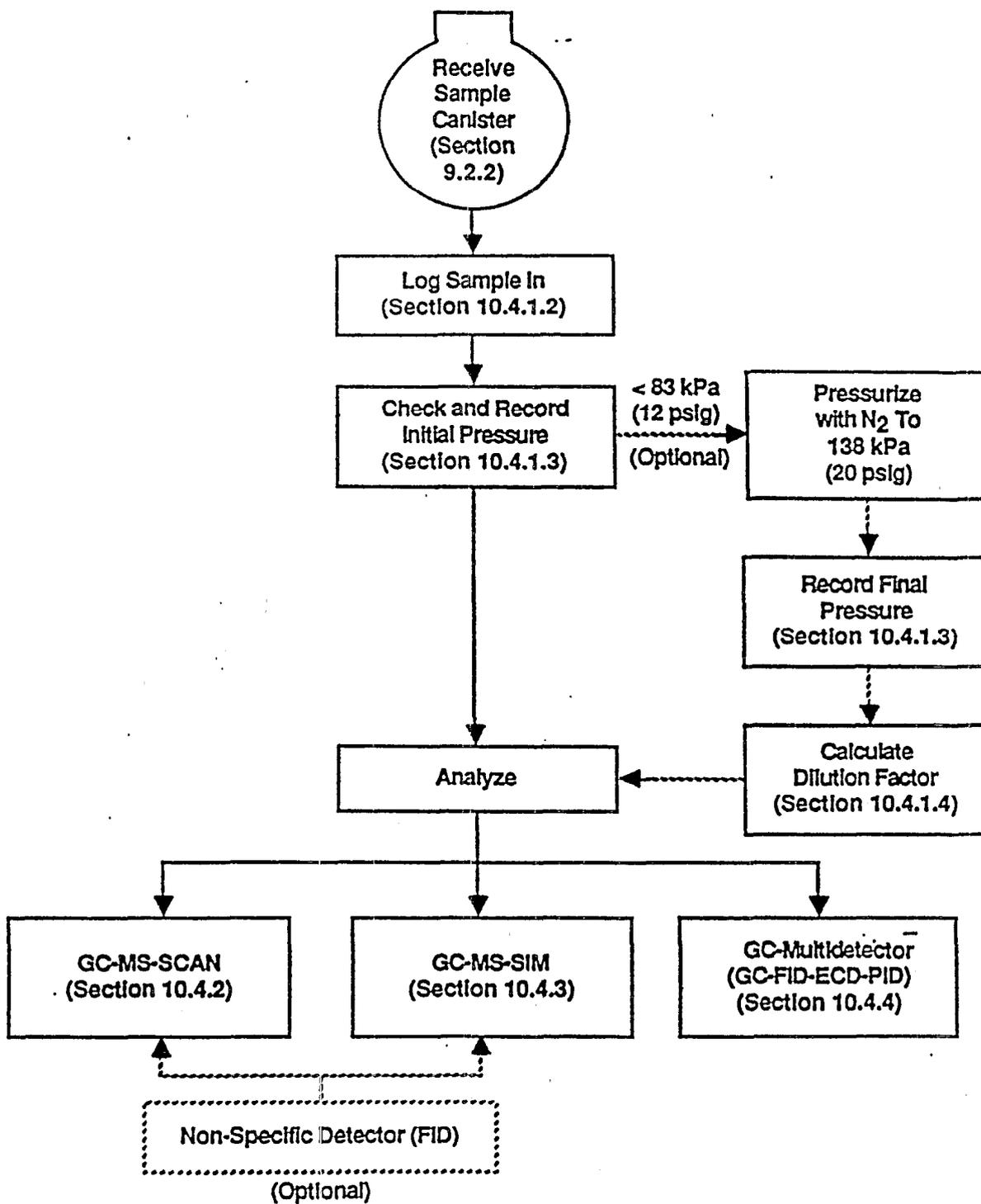


FIGURE 1. ANALYTICAL SYSTEMS AVAILABLE FOR CANISTER VOC IDENTIFICATION AND QUANTITATION

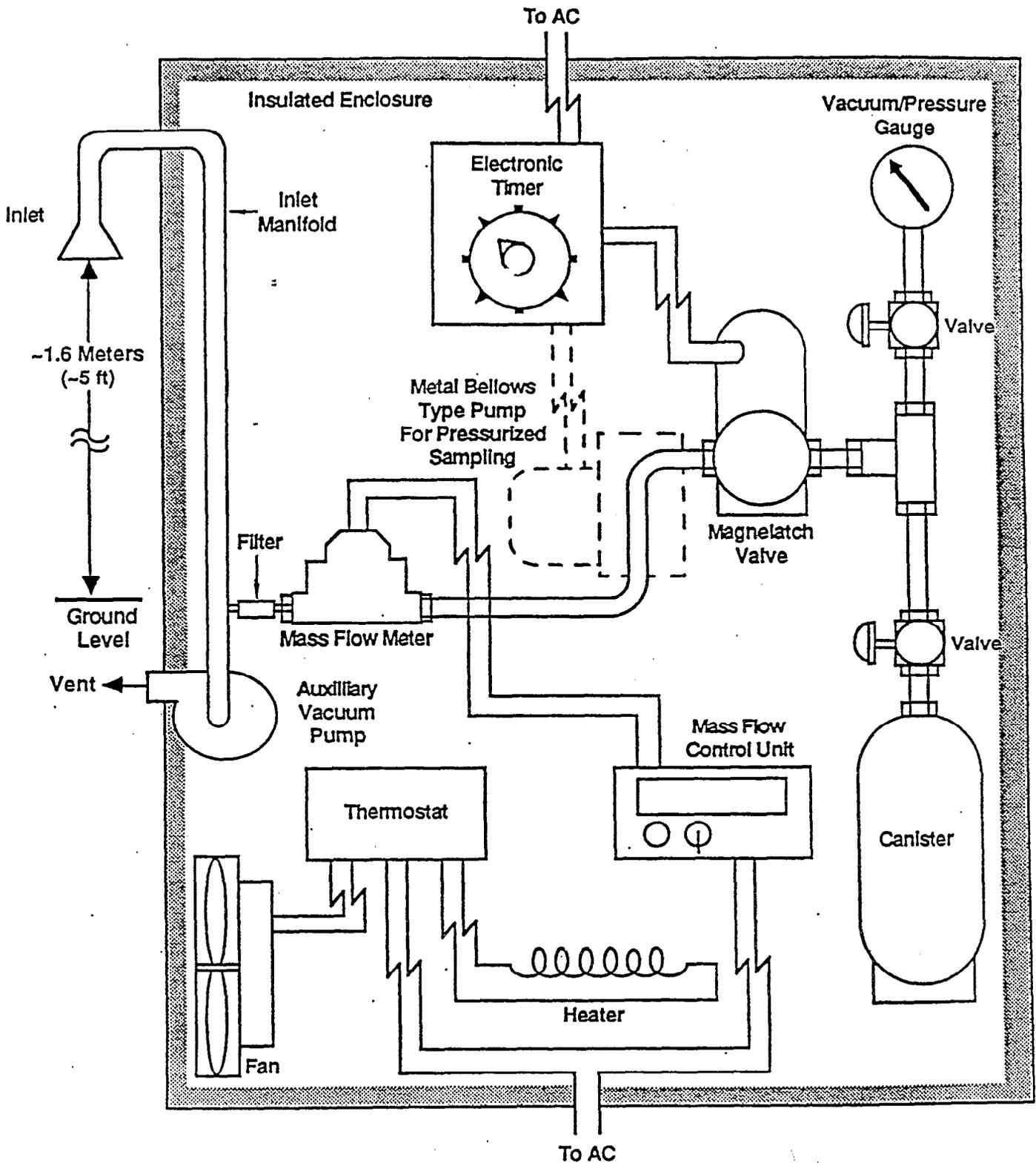


FIGURE 2. SAMPLER CONFIGURATION FOR SUBATMOSPHERIC PRESSURE OR PRESSURIZED CANISTER SAMPLING

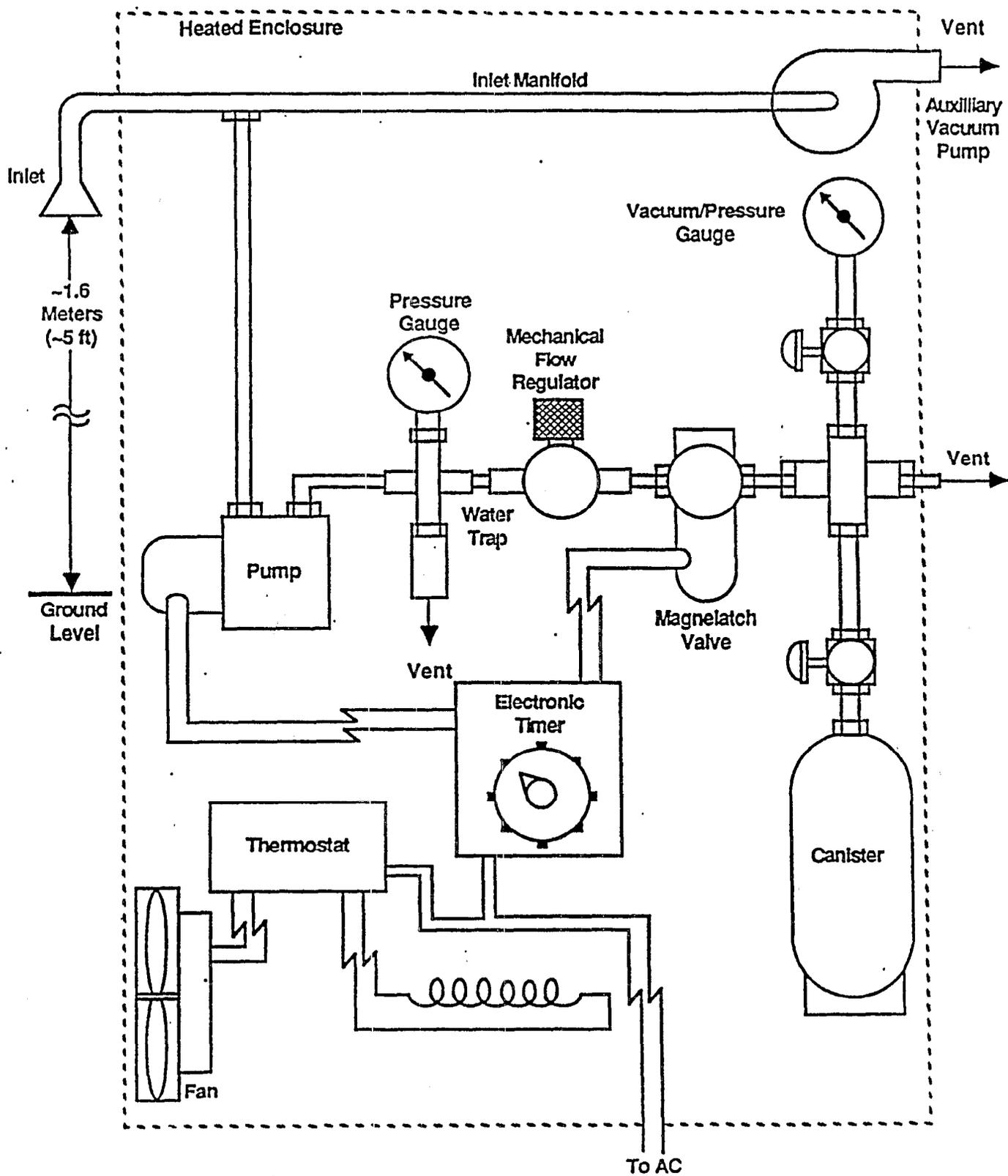
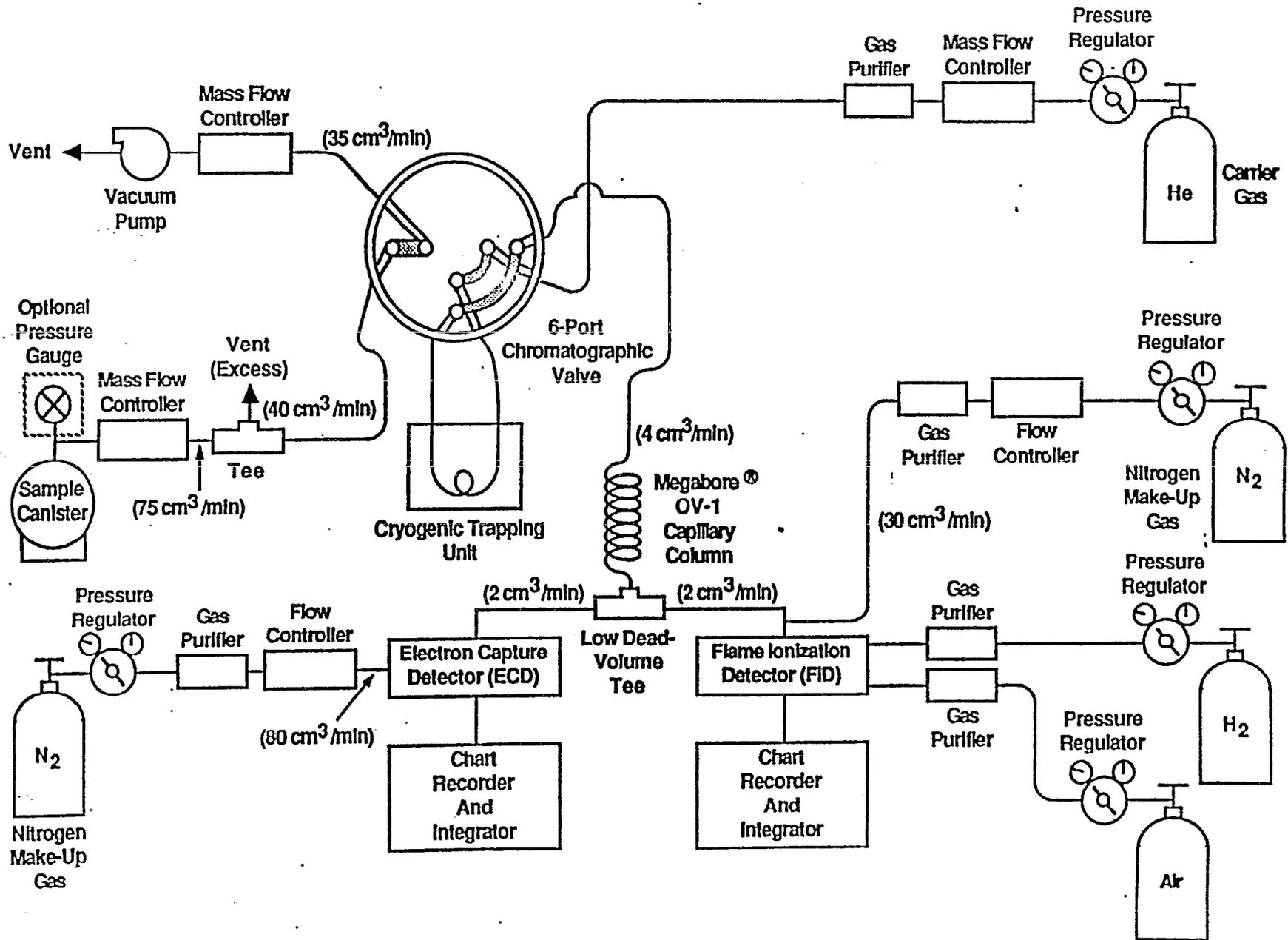


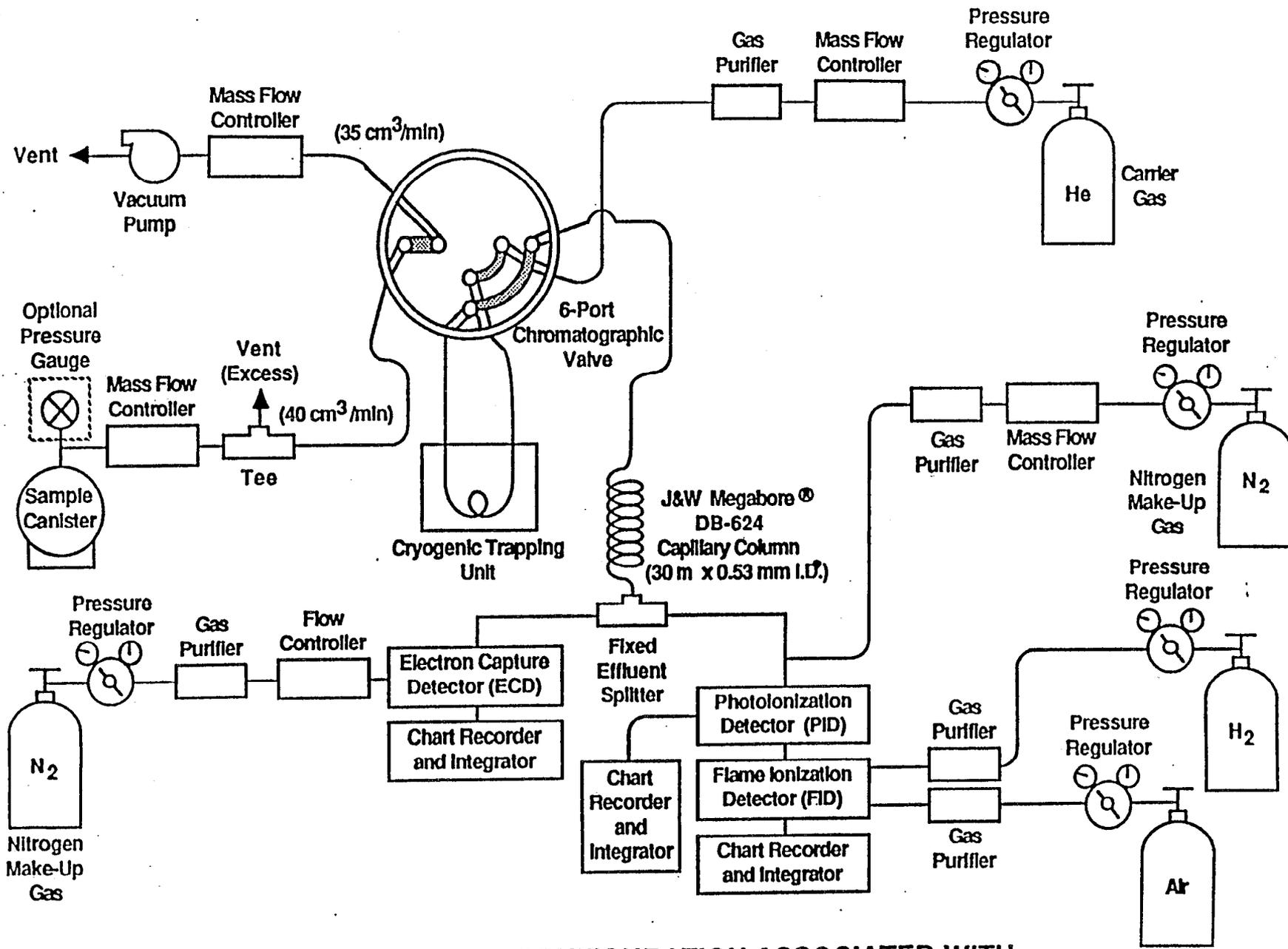
FIGURE 3. ALTERNATIVE SAMPLER CONFIGURATION FOR PRESSURIZED CANISTER SAMPLING





T014-78

FIGURE 5. GC-FID-ECD ANALYTICAL SYSTEM WITH THE 6-PORT CHROMATOGRAPHIC VALVE IN THE SAMPLE DESORPTION MODE



T014-79

**FIGURE 6. SYSTEM CONFIGURATION ASSOCIATED WITH THE GC-FID-ECD-PID ANALYTICAL SYSTEM WITH THE 6-PORT CHROMATOGRAPHIC VALVE IN THE SAMPLE DESORPTION MODE**

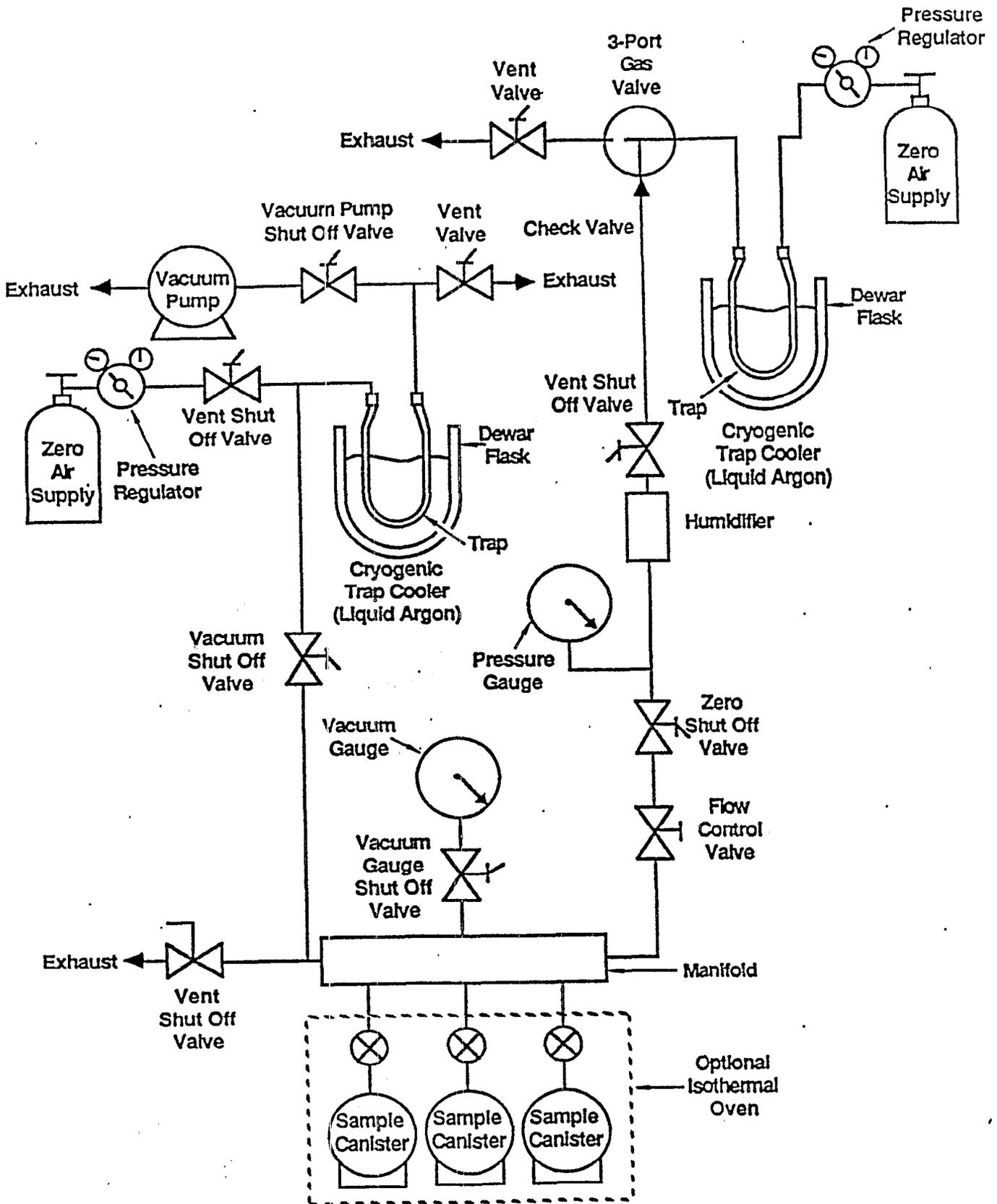
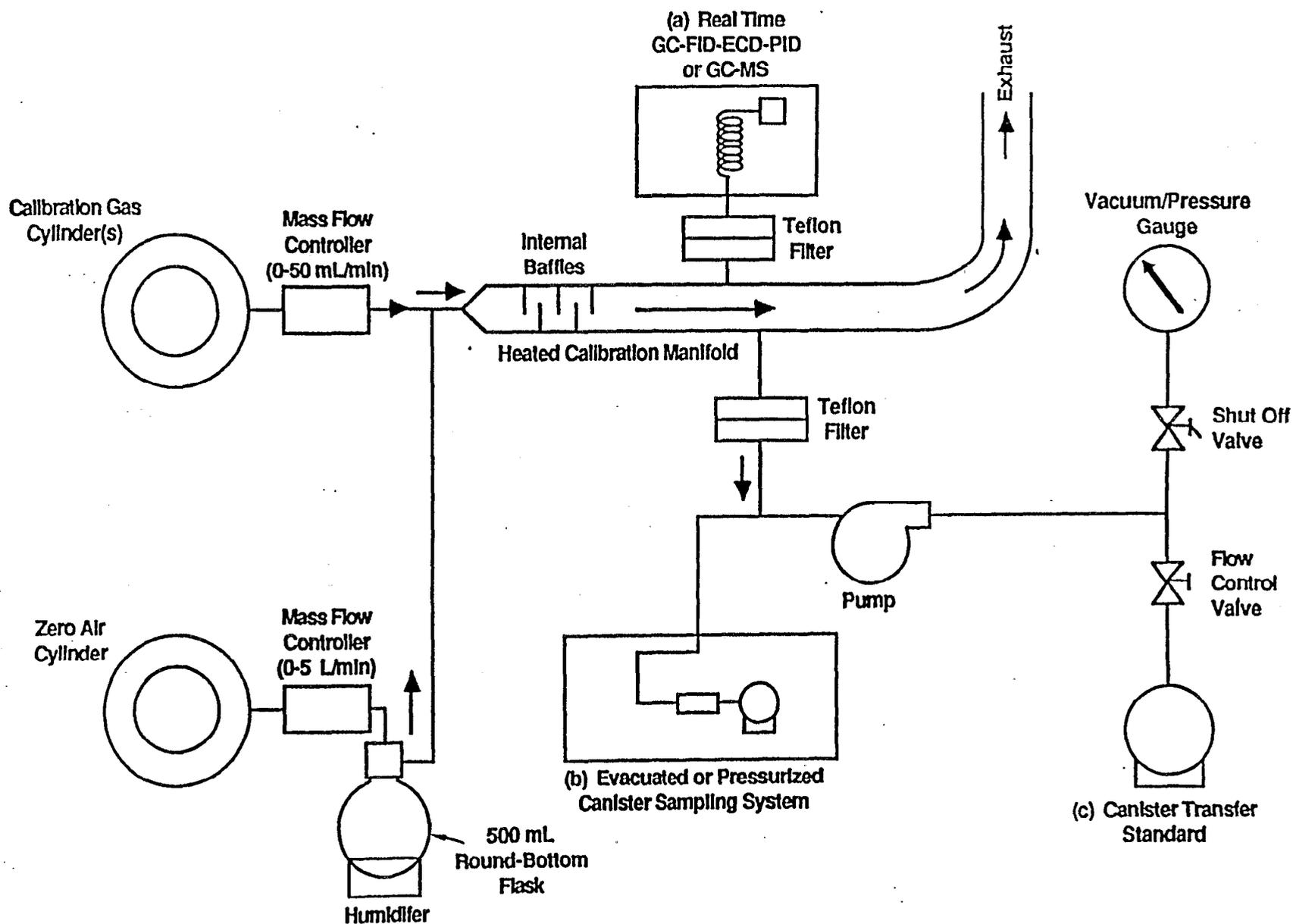
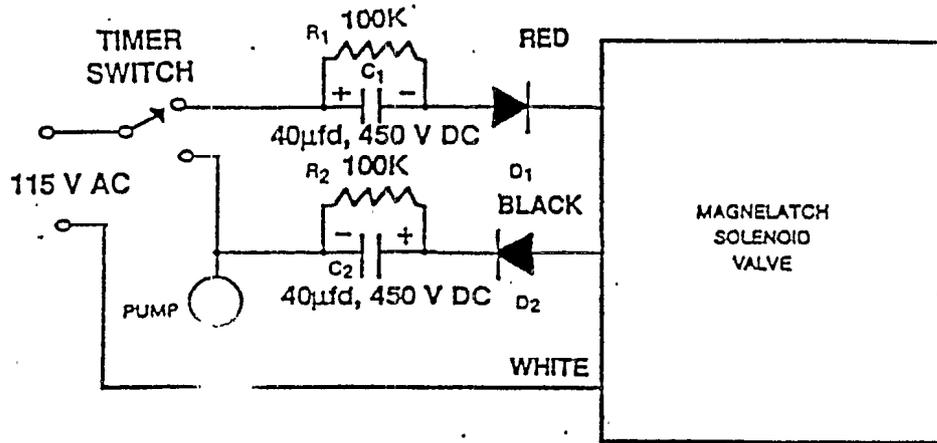


FIGURE 7. CANISTER CLEANING SYSTEM



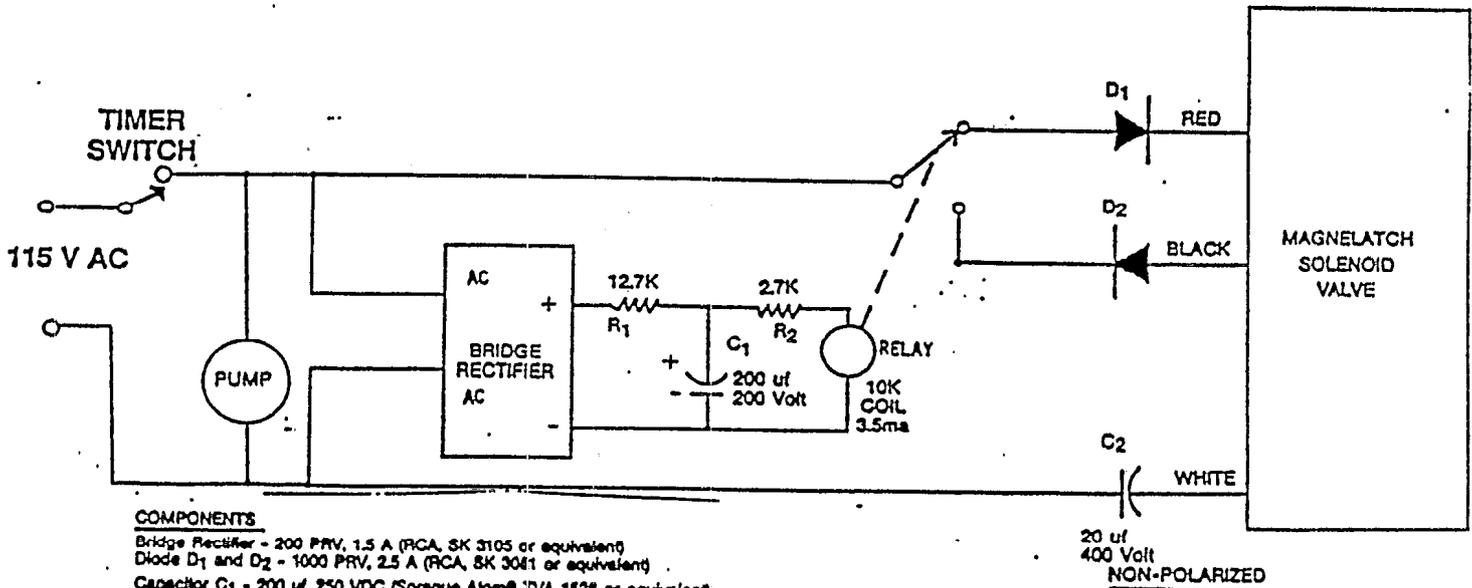
T014-81

FIGURE 8. SCHEMATIC OF CALIBRATION SYSTEM AND MANIFOLD FOR (a) ANALYTICAL SYSTEM CALIBRATION, (b) TESTING CANISTER SAMPLING SYSTEM AND (c) PREPARING CANISTER TRANSFER STANDARDS.



- COMPONENTS**  
 Capacitor C<sub>1</sub> and C<sub>2</sub> - 40 µf, 450 VDC (Sprague Atom® TVA 1712 or equivalent)  
 Resistor R<sub>1</sub> and R<sub>2</sub> - 0.5 watt, 5% tolerance  
 Diode D<sub>1</sub> and D<sub>2</sub> - 1000 PRV, 2.5 A (RCA, 81K 3061 or equivalent)

(a). Simple Circuit For Operating Magnelatch Valve



- COMPONENTS**  
 Bridge Rectifier - 200 PRV, 1.5 A (RCA, SK 3105 or equivalent)  
 Diode D<sub>1</sub> and D<sub>2</sub> - 1000 PRV, 2.5 A (RCA, SK 3061 or equivalent)  
 Capacitor C<sub>1</sub> - 200 µf, 250 VDC (Sprague Atom® TVA 1528 or equivalent)  
 Capacitor C<sub>2</sub> - 20 µf, 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)  
 Resistor R<sub>1</sub> and R<sub>2</sub> - 0.5 watt, 5% tolerance

(b). Improved Circuit Designed To Handle Power Interruptions

FIGURE 9. ELECTRICAL PULSE CIRCUITS FOR DRIVING SKINNER MAGNELATCH SOLENOID VALVE WITH A MECHANICAL TIMER

CANISTER SAMPLING FIELD 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			<del> </del>	<del> </del>
STOP				

PRESSURE	
CANISTER PRESSURE	
<del> </del>	

	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

DATE RECEIVED: \_\_\_\_\_  
 RECEIVED BY: \_\_\_\_\_  
 INITIAL PRESSURE: \_\_\_\_\_  
 FINAL PRESSURE: \_\_\_\_\_  
 DILUTION FACTOR: \_\_\_\_\_

ANALYSIS  
 GC-FID-ECD DATE: \_\_\_\_\_  
 GC-MSD-SCAN DATE: \_\_\_\_\_  
 GC-MSD-SIM DATE: \_\_\_\_\_

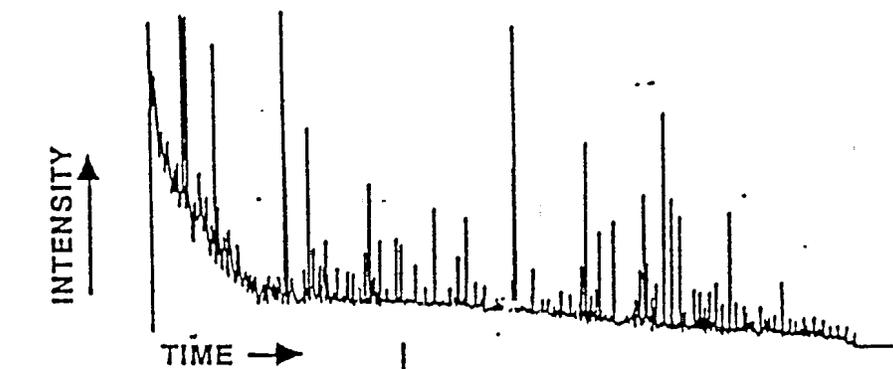
RESULTS\*: \_\_\_\_\_

GC-FID-ECD: \_\_\_\_\_  
 GC-MSD-SCAN: \_\_\_\_\_  
 GC-MSD-SIM: \_\_\_\_\_

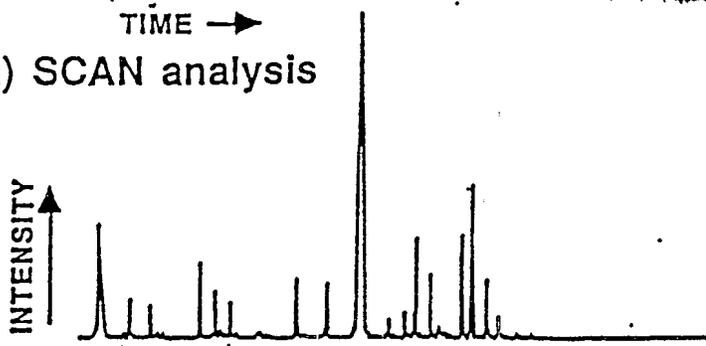
\_\_\_\_\_  
 SIGNATURE/TITLE

\* ATTACH DATA SHEETS

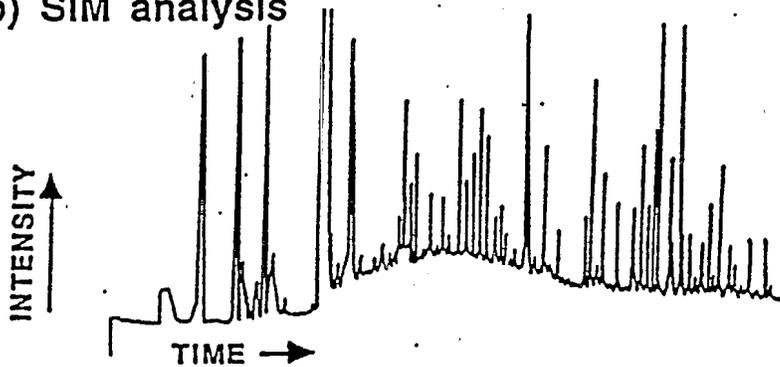
FIGURE 10. CANISTER SAMPLING FIELD DATA SHEET



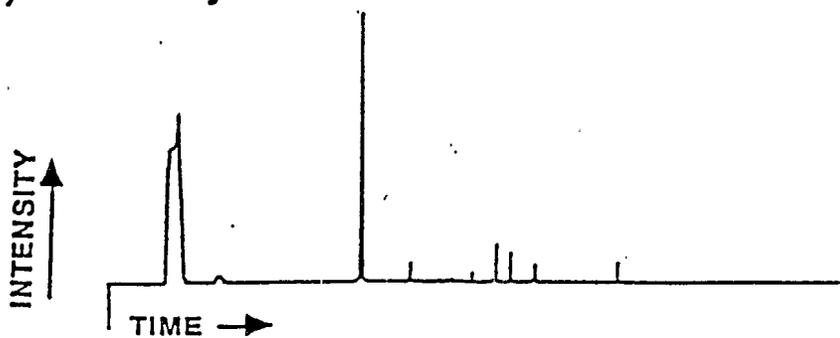
(a) SCAN analysis



(b) SIM analysis



(c) FID analysis



(d) ECD analysis

FIGURE 11. TYPICAL CHROMATOGRAMS OF A VOC SAMPLE ANALYZED BY GC-MS-SCAN-SIM MODE AND GC-MULTIDETECTOR MODE

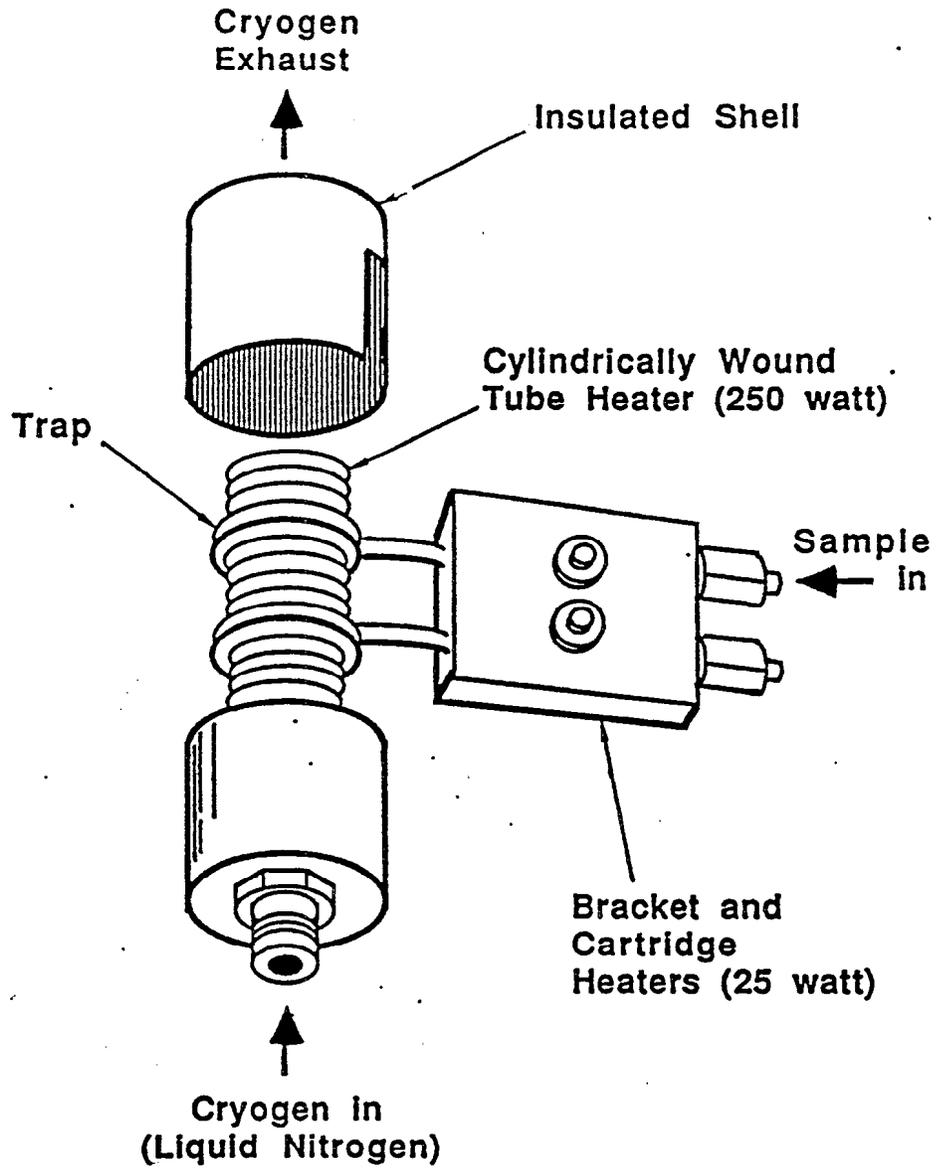


FIGURE 12. CRYOGENIC TRAPPING UNIT

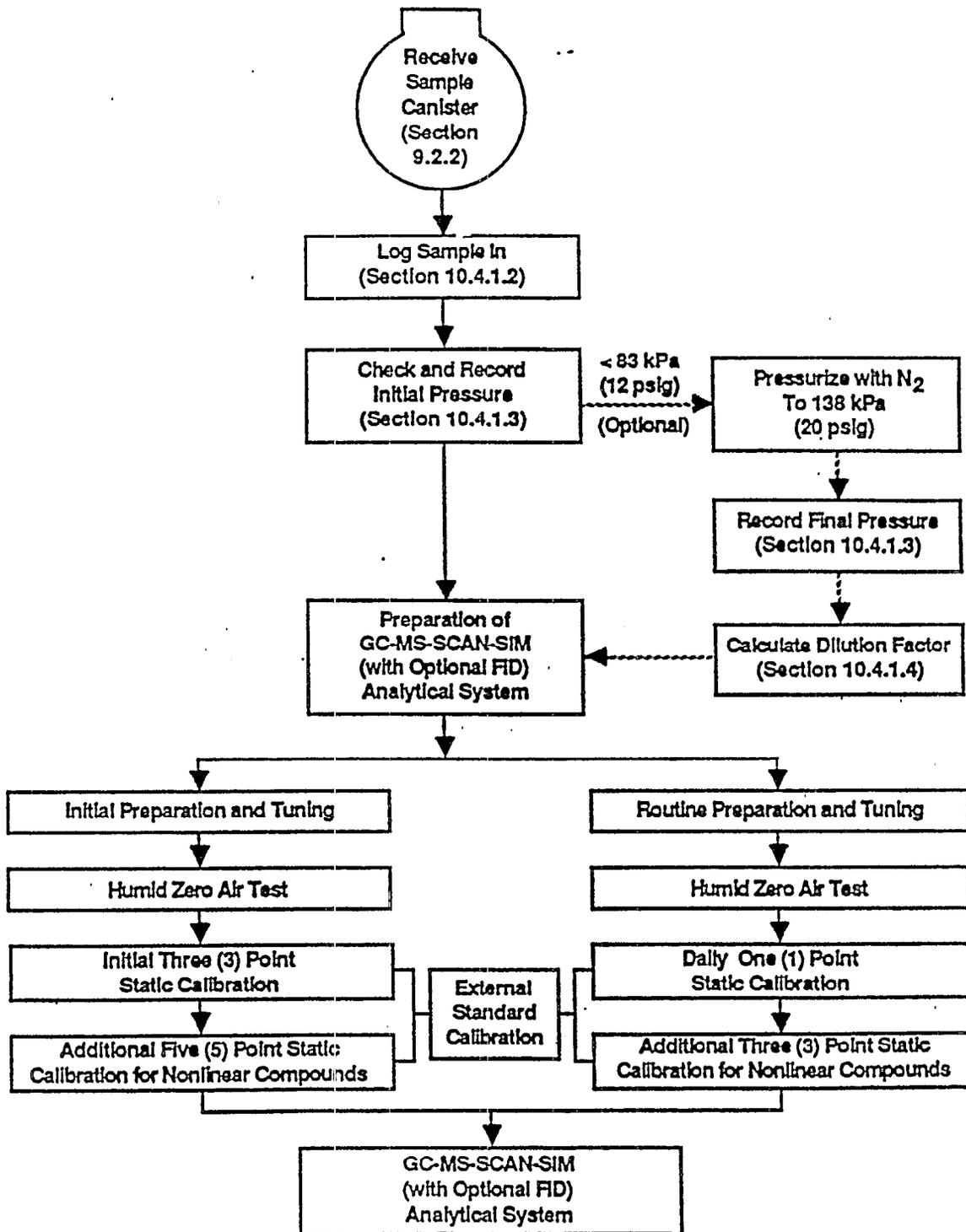


FIGURE 13. FLOWCHART OF GC-MS-SCAN-SIM ANALYTICAL SYSTEM PREPARATION (WITH OPTIONAL FID SYSTEM)

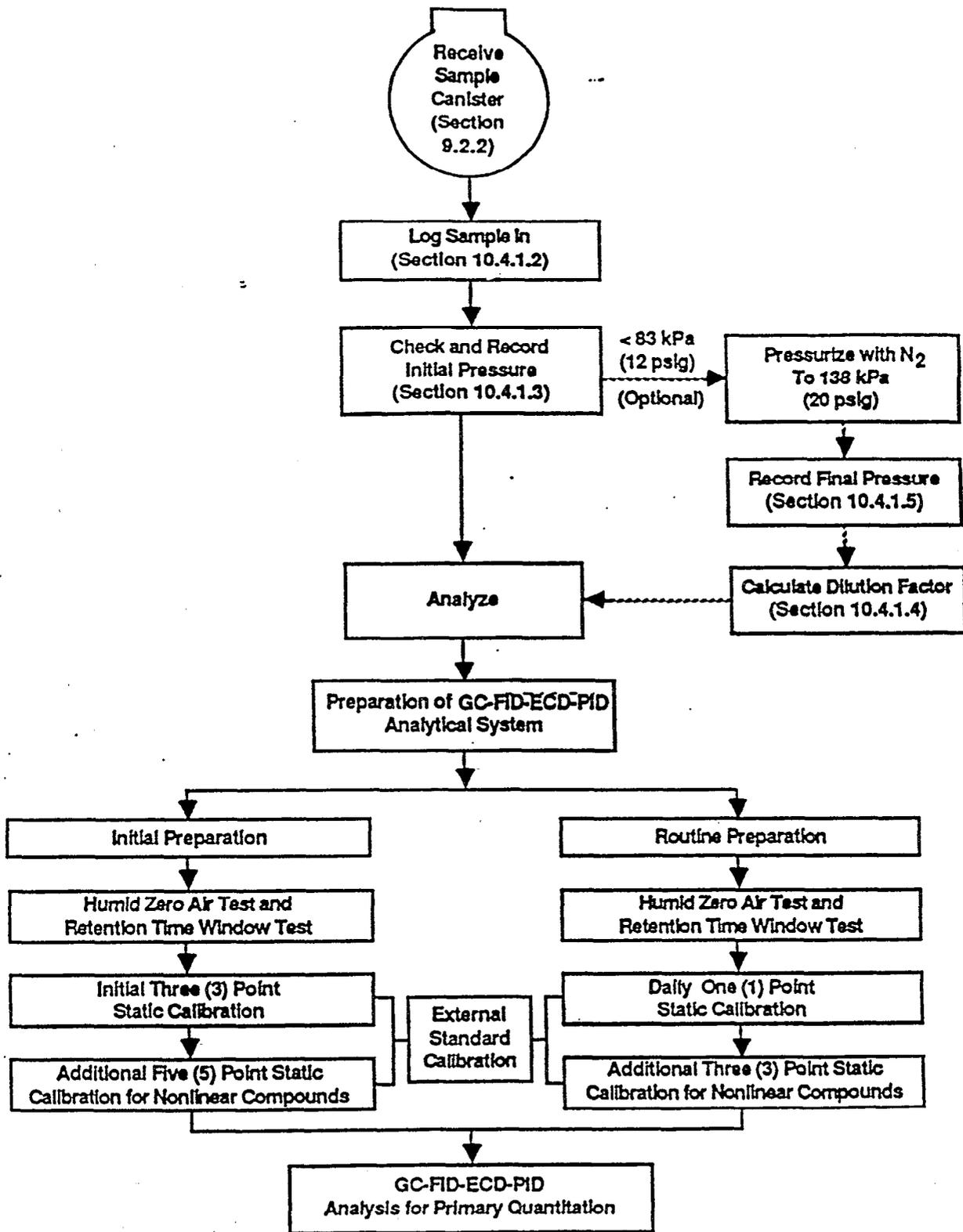
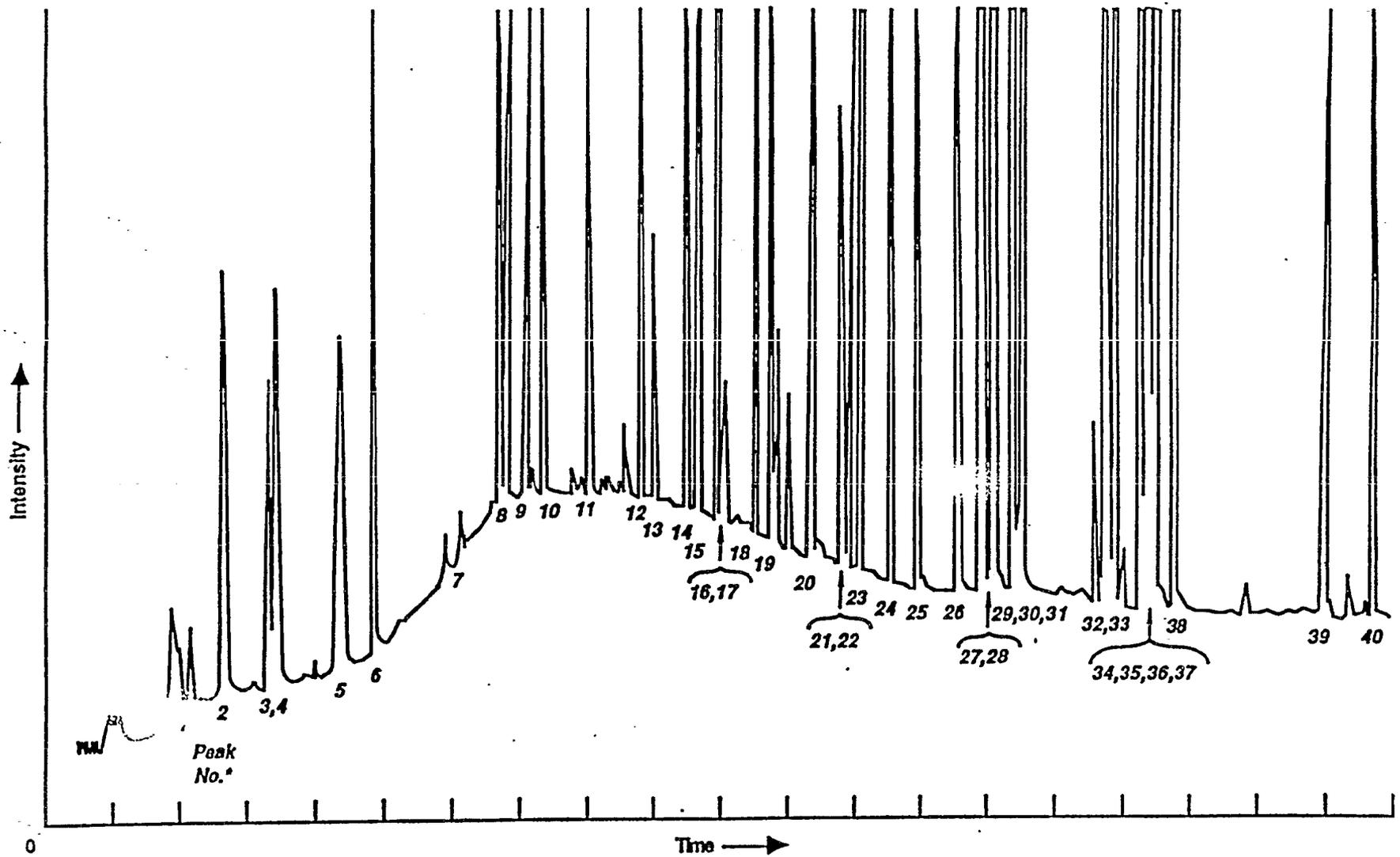


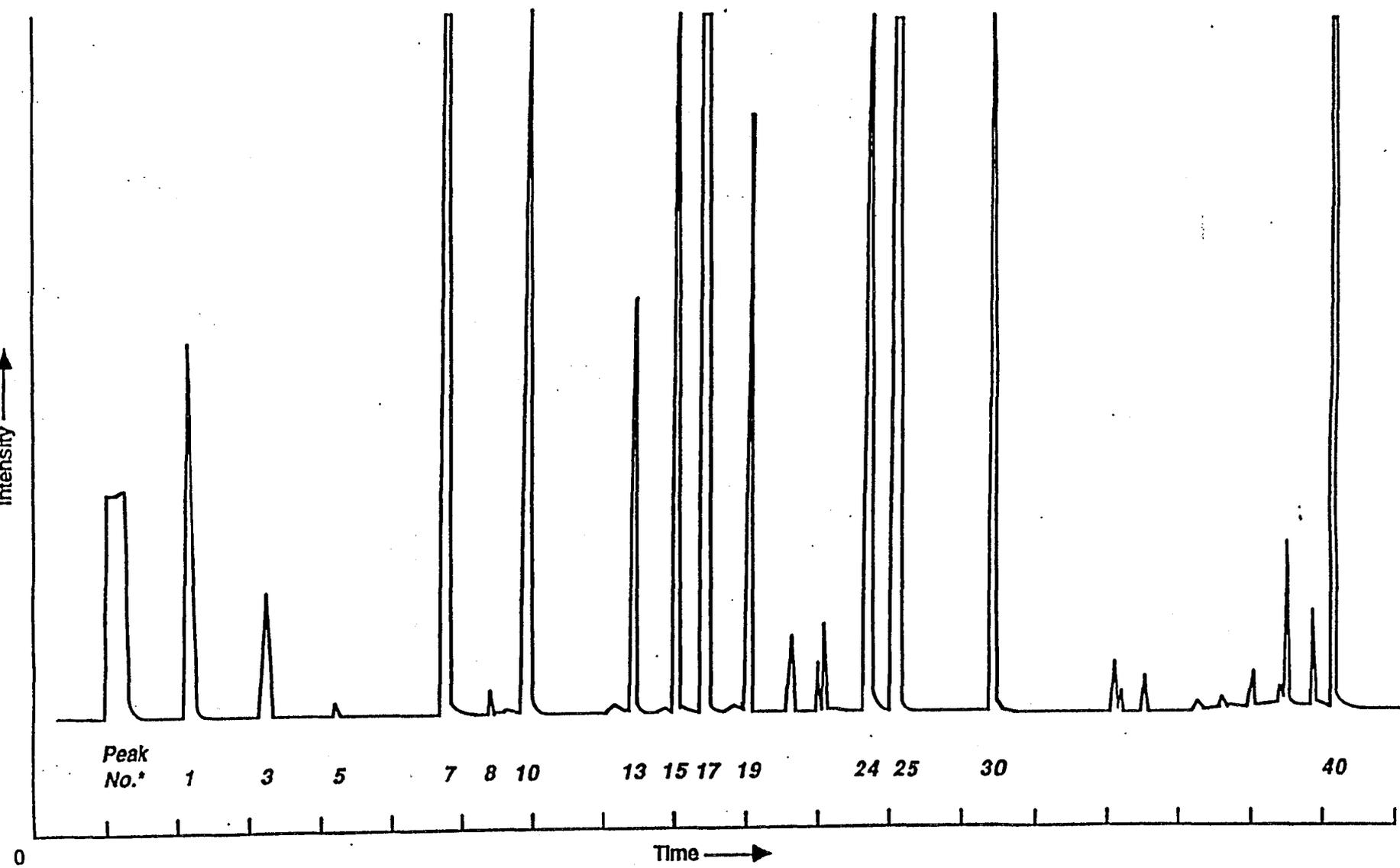
FIGURE 14. FLOWCHART OF GC-FID-ECD-PID ANALYTICAL SYSTEM PREPARATION



T014-88

\* See Table 7.0 For Peak Identification

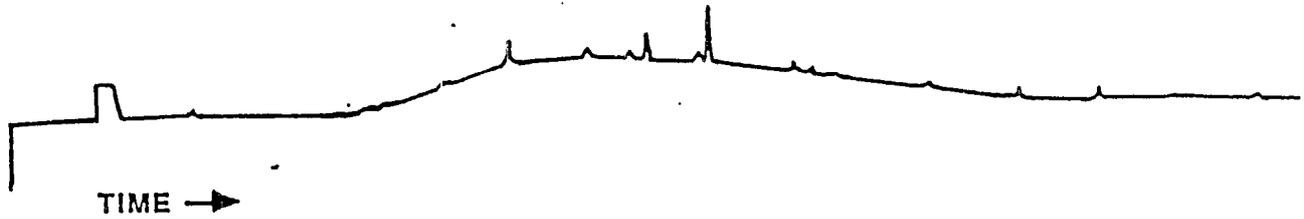
FIGURE 15. TYPICAL FID RESPONSE TO SELECTIVE VOCs



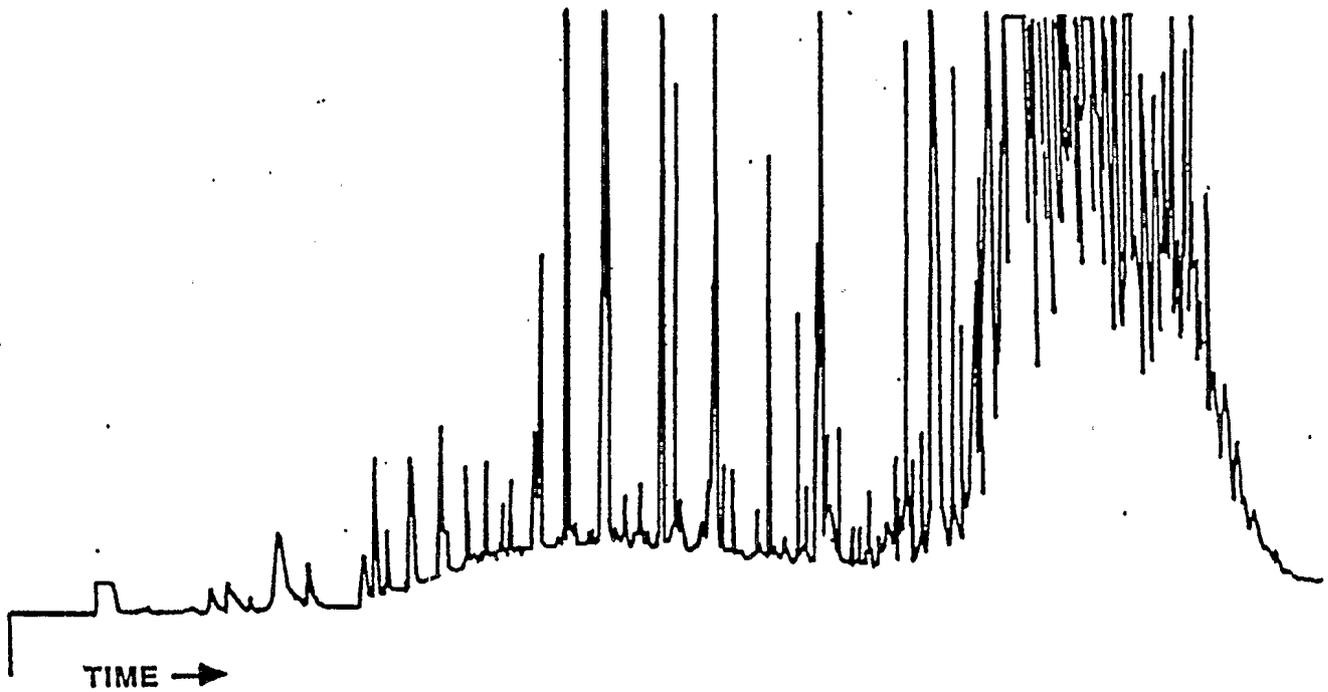
T014-89

\* See Table 7.0 For Peak Identification

FIGURE 16. TYPICAL ECD RESPONSE TO SELECTIVE VOCs



(a). Certified Sampler



(b). Contaminated Sampler

FIGURE 17. EXAMPLE OF HUMID ZERO AIR TEST RESULTS FOR A CLEAN SAMPLER (a) AND A CONTAMINATED SAMPLER (b)

T014-91

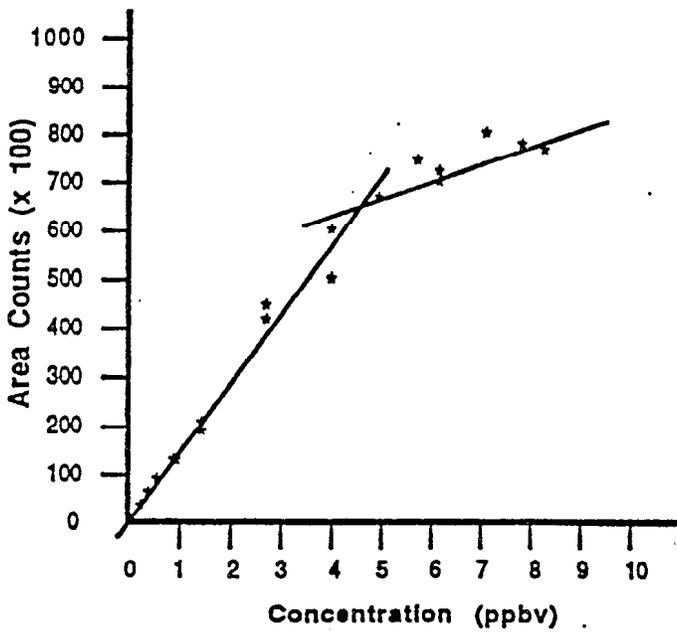


FIGURE 18(a). NONLINEAR RESPONSE OF TETRACHLOROETHYLENE ON THE ECD

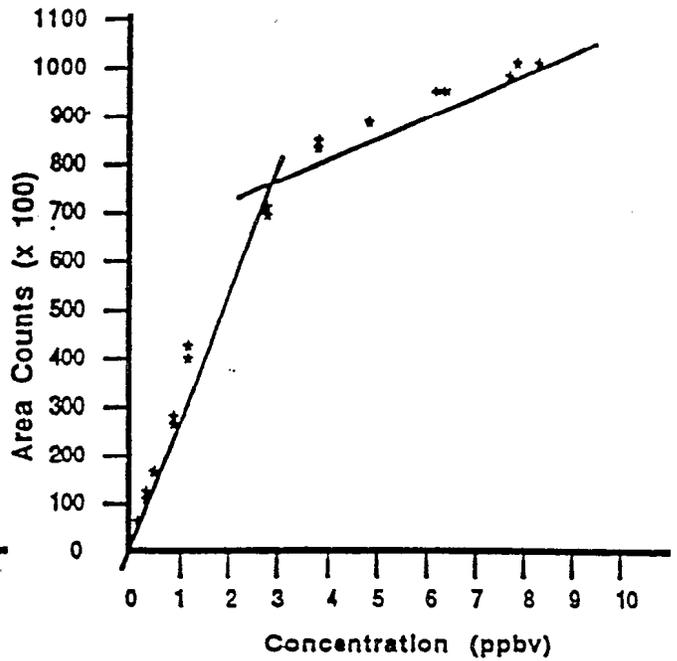


FIGURE 18(b). NONLINEAR RESPONSE OF CARBON TETRACHLORIDE ON THE ECD

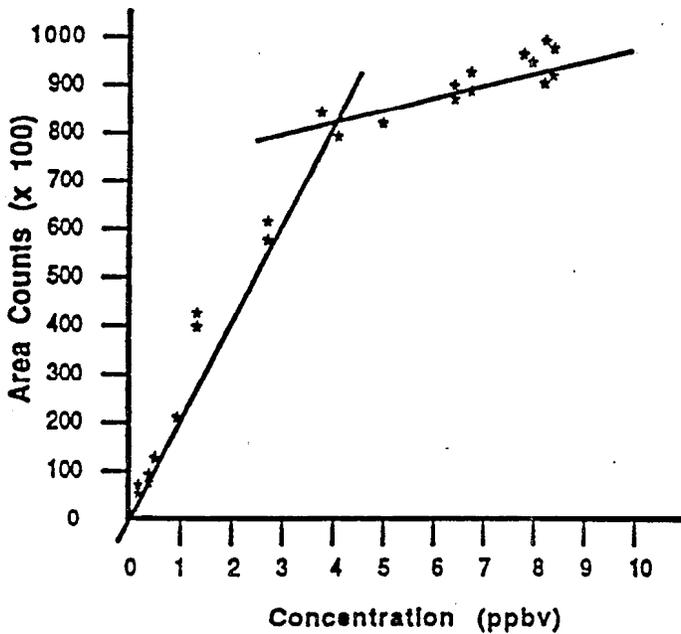


FIGURE 18(c). NONLINEAR RESPONSE OF HEXACHLOROBUTADIENE ON THE ECD

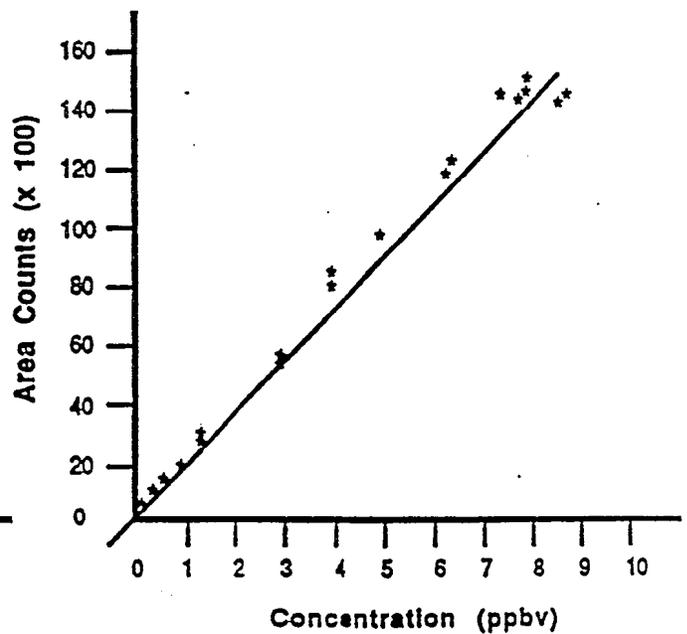
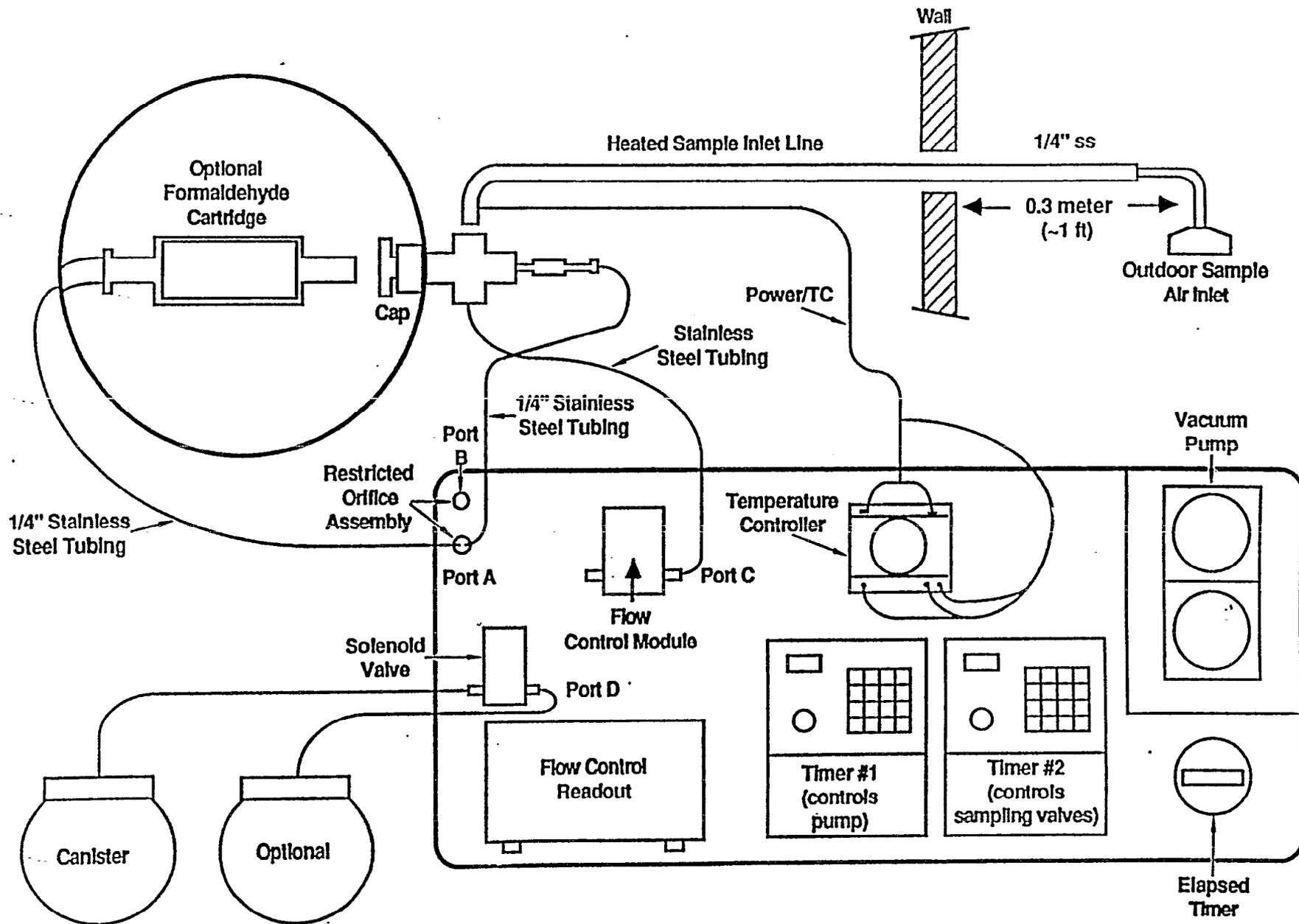


FIGURE 18(d). LINEAR RESPONSE OF CHLOROFORM ON THE ECD

FIGURE 18. RESPONSE OF ECD TO VARIOUS VOCs



T014-92

FIGURE 19. U.S. ENVIRONMENTAL PROTECTION AGENCY UATP, SCHEMATIC OF SAMPLE INLET CONNECTIONS SAMPLER

T014-93

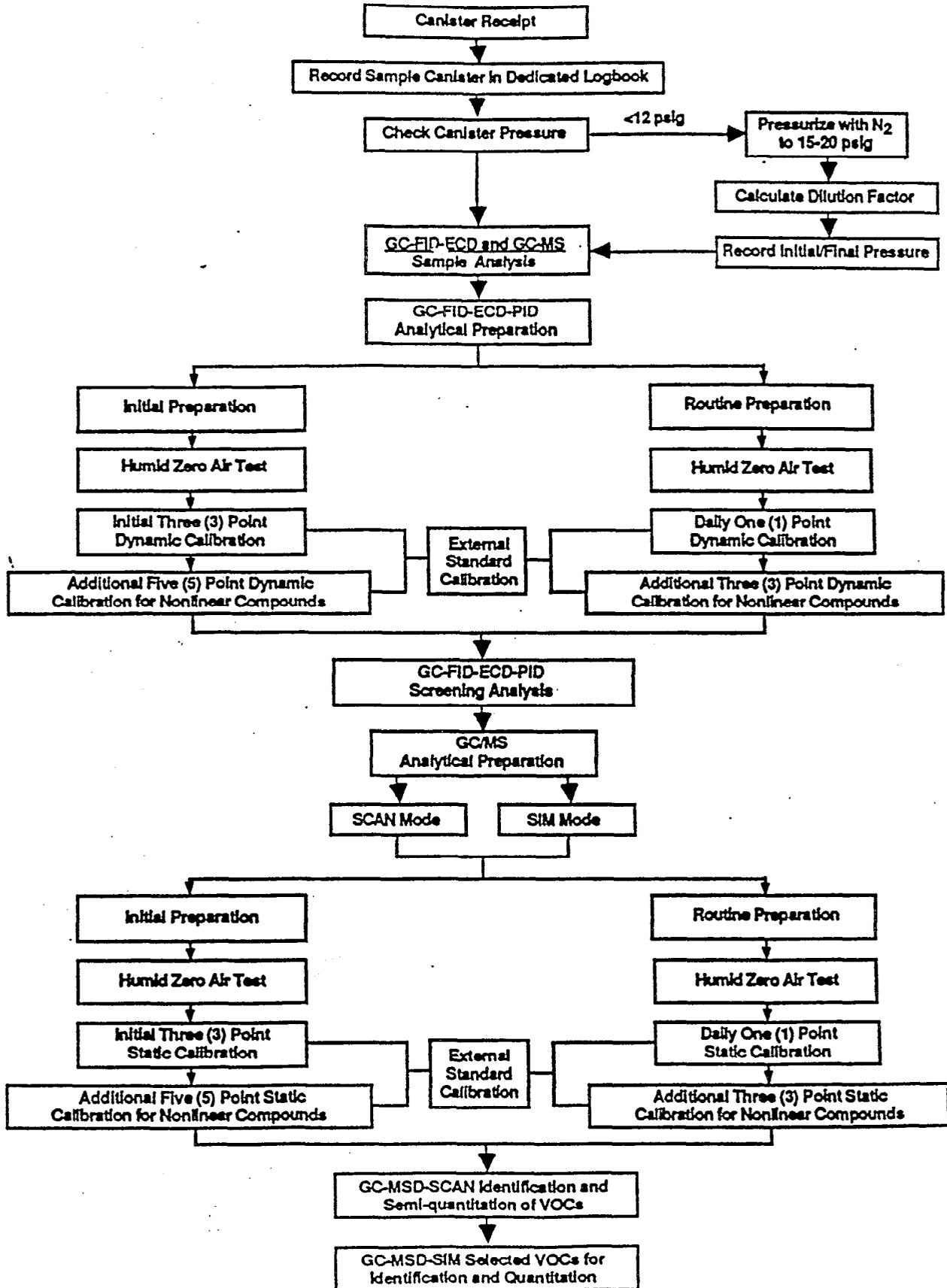
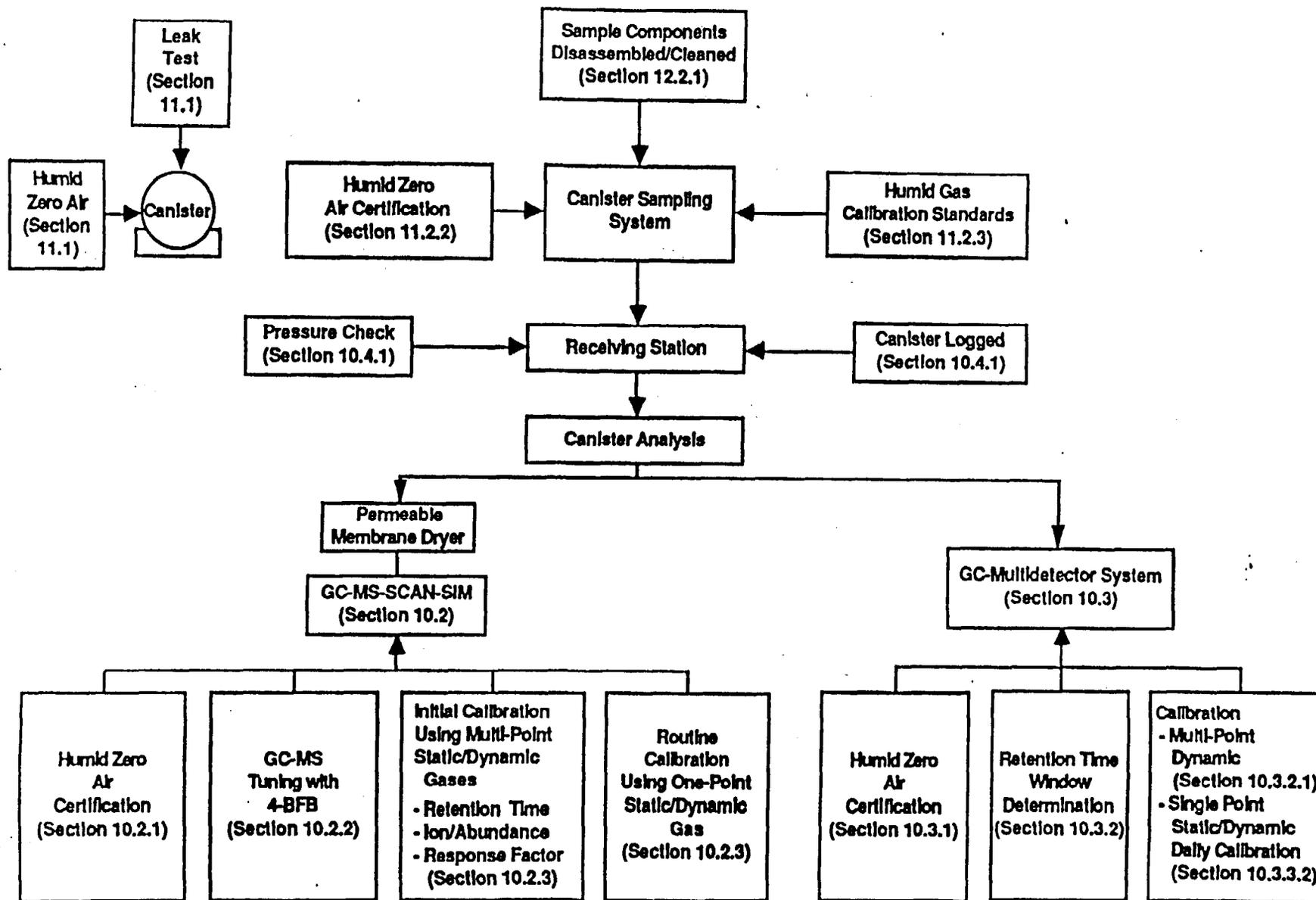


FIGURE 20. FLOWCHART OF ANALYTICAL SYSTEMS PREPARATION.



T014-94

**FIGURE 21. SYSTEM QUALITY ASSURANCE/QUALITY CONTROL (QA/QC) ACTIVITIES ASSOCIATED WITH VARIOUS ANALYTICAL SYSTEMS**

## APPENDIX A

### AVAILABILITY OF AUDIT CYLINDERS FROM UNITED STATES ENVIRONMENTAL PROTECTION AGENCY USEPA PROGRAMS/ REGIONAL OFFICES, STATE AND LOCAL AGENCIES AND THEIR CONTRACTORS

#### 1. Availability of Audit Cylinders

- 1.1 The USEPA has available, at no charge, cylinder gas standards of hazardous organic compounds at the ppb level that may be used to audit the performance of indoor air source measurement systems.
- 1.2 Each audit cylinder contains 5 to 18 hazardous organic compounds in a balance of N<sub>2</sub> gas. Audit cylinders are available in several concentration ranges. The concentration of each organic compound in the audit cylinder is within the range illustrated in Table A-1.

#### 2. Audit Cylinder Certification

- 2.1 All audit cylinders are periodically analyzed to assure that cylinder concentrations have remained stable.
- 2.2 All stability analyses include quality control analyses of ppb hazardous organic gas standards prepared by the National Bureau of Standards for USEPA.

#### 3. Audit Cylinder Acquisition

- 3.1 USEPA program/regional offices, state/local agencies, and their contractors may obtain audit cylinders (and an audit gas delivery system, if applicable) for performance audits during:
  - o RCRA Hazardous Waste Trial Burns For PHOC's; and
  - o Ambient/Indoor Air Measurement of Toxic Organics.
- 3.2 The audit cylinders may be acquired by contacting:

Robert L. Lampe  
U.S. Environmental Protection Agency  
Environmental Monitoring Systems Laboratory  
Quality Assurance Division  
MD-77B  
Research Triangle Park, NC 27711  
919-541-4531

TABLE A-1. AVAILABLE USEPA PERFORMANCE  
AUDIT CYLINDERS

<u>Group I Compounds</u>	<u>Group II Compounds</u>	<u>Group III Compounds</u>
Carbon tetrachloride Chloroform Perchloroethylene Vinyl chloride Benzene	Trichloroethylene 1,2-dichloroethane 1,2-dibromoethane Acetonitrile Trichlorofluoromethane (Freon-11) Dichlorodifluoromethane (Freon-12) Bromomethane Methyl ethyl ketone 1,1,1-trichloroethane	Pyridine (Pyridine in Group III cylinders but certified analysis not available) Vinylidene chloride 1,1,2-trichloro-1,2,2-trifluoroethane (Freon-113) 1,2-dichloro-1,1,2,2-tetrafluoroethane (Freon-114) Acetone 1-4 Dioxane Toluene Chlorobenzene.
<u>Group I Ranges</u>	<u>Group II Ranges</u>	<u>Group III Ranges</u>
7 to 90 ppb 90 to 430 ppb 430 to 10,000 ppb	7 to 90 ppb 90 to 430 ppb	7 to 90 ppb 90 to 430 ppb
<u>Group IV</u>	<u>Group V</u>	
Acrylonitrile 1,3-butadiene Ethylene oxide Methylene chloride Propylene oxide o-xylene	Carbon tetrachloride Chloroform Perchloroethylene Vinyl chloride Benzene Trichloroethylene 1,2-dichloroethane 1,2-dibromoethane 1,1,1-trichloroethane	Methylene chloride Trichlorofluoromethane (Freon-11) Bromomethane Toluene Chlorobenzene 1,3-Butadiene o-xylene Ethyl benzene 1,2-dichloropropane
<u>Group IV Ranges</u>	<u>Group V Ranges</u>	
7 to 90 ppb 430 to 10,000 ppb	1 to 40 ppb	

## APPENDIX B

### OPERATING PROCEDURES FOR A PORTABLE GAS CHROMATOGRAPH EQUIPPED WITH A PHOTOIONIZATION DETECTOR

#### 1. Scope

This procedure is intended to screen ambient air environments for volatile organic compounds. Screening is accomplished by collection of VOC samples within an area and analysis onsite using a portable gas chromatograph/integrator (Photovac Models 10S10, 10S50) or equivalent. This procedure is not intended to yield quantitative or definite qualitative information regarding the substances detected. Rather, it provides a chromatographic "profile" of the occurrence and intensity of unknown volatile compounds which assists in placement of fixed-site samplers.

#### 2. Applicable Documents

##### 2.1 ASTM Standards

E260 - Recommended Practice for General Gas Chromatography Procedures

E355 - Practice for Gas Chromatography Terms and Relationships

##### 2.2 Other Documents

Portable Instruments User's Manual for Monitoring VOC Sources,  
EPA-34011-86-015, U.S. Environmental Protection Agency, Washington, DC, June, 1986.

#### 3. Summary of Method

3.1 An air sample is extracted directly from ambient air and analyzed on-site by a portable GC.

3.2 Analysis is accomplished by drawing an accurate volume of ambient air through a sampling port and into a concentrator, then the sample air is transported by carrier gas onto a packed column and into a PID, resulting in response peak(s). Retention times are compared with those in a standard chromatogram to predict the probable identity of the sample components.

#### 4. Significance

4.1 VOCs are emitted into the atmosphere from a variety of sources including petroleum refineries, synthetic organic chemical plants,

natural gas processing plants, and automobile exhaust. Many of these VOC emissions are acutely toxic; therefore, their determination in indoor air is necessary to assess human health impacts.

- 4.2 Conventional methods for VOC determination use solid sorbent and canister sampling techniques.
- 4.3 Collection of indoor air samples in canisters provides (1) convenient integration of ambient samples over a specific time period, (e.g., 24 hours); (2) remote sampling and central analysis; (3) ease of storing and shipping samples, if necessary; (4) unattended sample collection; (5) analysis of samples from multiple sites with one analytical system; and (6) collection of sufficient sample volume to allow assessment of measurement precision and/or analysis of samples by several analytical systems.
- 4.4 The use of portable GC equipped with multidetectors has assisted air toxics programs by using the portable GC as a "screening tool" to determine "hot spots," potential interferences, and semi-quantitation of VOCs/SVOCs, prior to locating more traditional fixed-site samplers.

## 5. Definitions

Definitions used in this document and in any user-prepared Standard Operating Procedures (SOPs) should be consistent with ASTM Methods D1356 and E355. Abbreviations and symbols pertinent to this method are defined at point of use.

## 6. Interferences

- 6.1 The most significant interferences result from extreme differences in limits of detection (LOD) among the target VOCs (Table B-1). Limitations in resolution associated with ambient temperature, chromatography and the relatively large number of chemicals result in coelution of many of the target components. Coelution of compounds with significantly different PID sensitivities will mask compounds with more modest sensitivities. This will be most dramatic in interferences from benzene and toluene.

- 6.2 A typical chromatogram and peak assignments of a standard mixture of target VOCs (under the prescribed analytical conditions of this method) are illustrated in Figure B-1. Samples which contain a highly complex mixture of components and/or interfering levels of benzene and toluene are analyzed on a second, longer chromatographic column. The same liquid phase in the primary column is contained in the alternate column but at a higher percent loading.
- 6.3 Recent designs in commercially available GCs (Table B-2) have pre-concentrator capabilities for sampling lower concentrations of VOCs, pre-column detection with back-flush capability for shorter analytical time, constant column temperature for method precision and accuracy and multidetector (PID, ECD, and FID) capability for versatility. Many of these newer features address the weaknesses and interferences mentioned above.

## 7. Apparatus

- 7.1 Gas chromatograph. A GC (Photovac Inc., 739 B Parks Ave, Huntington, NY, 11743, Model 10S10 or 10S50), or equivalent used for surveying ambient air environments (which could employ a multidetector) for sensing numerous VOCs compounds eluting from a packed column at room temperatures. This particular portable GC procedure is written employing the photoionization detector as its major sensing device, as part of the Photovac Model 10S10 portable GC survey tool. Chromatograms are developed on a column of 3% SP-2100 on 100/120 Supelcoport (0.66 m x 3.2 mm I.D.) with a flow of 30 cm<sup>3</sup>/min air.
- 7.2 GC accessories. In addition to the basic gas chromatograph, several other pieces of equipment are required to execute the survey sampling. Those include gas-tight syringes for standard injection, alternate carrier gas supplies, high pressure connections for filling the internal carrier gas reservoir, and if the Model 10S10 is used, a recording integrator (Hewlett Packard, Avondale, PA, Model 3390A), or equivalent.

## 8. Reagents and Materials

- 8.1 Carrier gas. "Zero" air [ $<0.1$  ppm total hydrocarbon (THC)] is used as the carrier gas. This gas is conveniently contained in 0.84 m<sup>3</sup> (30 ft<sup>3</sup>) aluminum cylinders. Carrier gas of poorer quality

may result in spurious peaks in sample chromatograms. A Brooks, Type 1355-00F1AAA rotameter (or equivalent) with an R-215-AAA tube and glass float is used to set column flow.

- 8.2 System performance mixture. A mixture of three target compounds (e.g., benzene, trichloroethylene, and styrene) in nitrogen is used for monitoring instrument performance. The approximate concentration for each of the compounds in this mixture is 10 parts per billion (ppb). This mixture is manufactured in small, disposable gas cylinders [at 275 kPa (40 psi)] from Scott Specialty Gases, or equivalent.
- 8.3 Reagent grade nitrogen gas. A small disposable cylinder of high purity nitrogen gas is used for blank injections.
- 8.4 Sampling syringes. Gas-tight syringes, without attached shut-off valves (Hamilton Model 1002LT), or equivalent are used to introduce accurate sample volumes into the high pressure injectors on the portable gas chromatograph. Gas syringes with shut-off valves are not recommended because of memory problems associated with the valves. For samples suspected of containing high concentrations of volatile compounds, disposable glass syringes (e.g., Glaspak, or equivalent) with stainless steel/Teflon<sup>®</sup> hub needles are used.
- 8.5 High pressure filler. An adapter (Photovac SA101, or equivalent) for filling the internal carrier gas reservoir on the portable GC is used to deliver "zero" air.

## 9. Procedure

### 9.1 Instrument Setup

- 9.1.1 The portable gas chromatograph must be prepared prior to use in the ambient survey sampling. The pre-sampling activities consist of filling the internal carrier gas cylinder, charging the internal power supply, adjusting individual column carrier gas flows, and stabilizing the photoionization detector.
- 9.1.2 The internal reservoir is filled with "zero" air. The internal 12V, 6AH lead/acid battery can be recharged to provide up to eight hours of operation. A battery

which is discharged will automatically cause the power to the instrument to be shut down and will require an overnight charge. During AC operation, the batteries will automatically be trickle-charged or in a standby mode.

- 9.1.3 The portable GC should be operated (using the internal battery power supply) at least forty minutes prior to collection of the first sample to insure that the photoionization detector has stabilized. Upon arriving at the area to be sampled, the unit should be connected to AC power, if available.

## 9.2 Sample Collection

- 9.2.1 After the portable gas chromatograph is located and connected to 110V AC, the carrier gas flows must be adjusted. Flows to the 1.22 meter, 5% SE-30 and 0.66 meter, 3% SP2100 columns are adjusted with needle valves. Flows of 60 cm<sup>3</sup>/min (5% SE-30) and 30 cm<sup>3</sup>/min (3% SP2100) are adjusted by means of a calibrated rotameter. Switching between the two columns is accomplished by turning the valve located beneath the electronic module. During long periods of inactivity, the flows to both columns should be reduced to conserve pressure in the internal carrier gas supply. The baseline on the recorder/integrator is set to 20% full scale.
- 9.2.2 Prior to analysis of actual samples, an injection of the performance evaluation mixture must be made to verify chromatographic and detector performance. This is accomplished by withdrawing 1.0 mL samples of this mixture from the calibration cylinder and injecting it onto the SP2100 column. The next sample analyzed should be a blank, consisting of reagent grade nitrogen.
- 9.2.3 Ambient air samples are injected onto the 3% SP2100 column. The chromatogram is developed for 15 minutes. Samples which produce particularly complex chromatograms

especially for early eluting components, are reinjected on the 5% SE-30 column. [Note: In no instance should a syringe which has been used for the injection of the calibrant/system performance mixture be used for the acquisition and collection of samples, or vice versa.]

- 9.2.4 Samples have generally been collected from the ambient air at sites which are near suspected sources of VOCs and SVOCs and compared with those which are not. Typically, selection of sample locations is based on the presence of chemical odors. Samples collected in areas without detectable odors have not shown significant PID responses. Therefore, sampling efforts should be initially concentrated on "suspect" environments (i.e., those which have appreciable odors). The objective of the sampling is to locate sources of the target compounds. Ultimately, samples should be collected throughout the entire location, but with particular attention given to areas of high or frequent occupation.

### 9.3 Sample Analysis

- 9.3.1 Qualitative analysis. Positive identification of sample components is not the objective of this "screening" procedure. Visual comparison of retention times to those in a standard chromatogram (Figure B-1) are used only to predict the probable sample component types.
- 9.3.2 Estimation of levels. As with qualitative analysis, estimates of component concentrations are extremely tentative and are based on instrument responses to the calibrant species (e.g., benzene, trichloroethylene, styrene), the proposed component identification, and the difference in response between sample component and calibrant. For purposes of locating pollutant emission sources, roughly estimated concentrations and suspected compound types are considered sufficient.

## 10. Performance Criteria and Quality Assurance

Required quality assurance measures and guidance concerning performance criteria that should be achieved within each laboratory are summarized and provided in the following section.

### 10.1 Standard Operating Procedures

10.1.1 SOPs should be generated by the users to describe and document the following activities in their laboratory: (1) assembly, calibration, leak check, and operation of the specific portable GC sampling system and equipment used; (2) preparation, storage, shipment, and handling of the portable GC sampler; (3) purchase, certification, and transport of standard reference materials; and (4) all aspects of data recording and processing, including lists of computer hardware and software used.

10.1.2 Specific stepwise instructions should be provided in the SOPs and should be readily available to and understood by the personnel conducting the survey work.

### 10.2 Quality Assurance Program

10.2.1 Reagent and materials control. The carrier gas employed with the portable GC is "zero air" containing less than 0.1 ppm VOCs. System performance mixtures are certified standard mixtures purchased from Scott Specialty Gases, or equivalent.

10.2.2 Sampling protocol and chain of custody. Sampling protocol sheets must be completed for each sample. Specifics of the sample with regard to sampling location, sample volume, analysis conditions, and supporting calibration and visual inspection information are detailed by these documents. An example form is exhibited in Table B-3.

#### 10.2.3 Blanks, Duplicates, and System Performance Samples

10.2.3.1 Blanks and Duplicates. Ten percent of all injections made to the portable GC are blanks,

where the blank is reagent grade nitrogen gas. This is the second injection in each sampling location. An additional 10% of all injections made are duplicate injections. This will enhance the probability that the chromatogram of a sample reflects only the composition of that sample and not any previous injection. Blank injections showing a significant amount of contaminants will be cause for remedial action.

10.2.3.2 System Performance Mixture. An injection of the system performance mixture will be made at the beginning of a visit to a particular sampling location (i.e., the first injection). The range of acceptable chromatographic system performance criteria and detector response is shown in Table B-4. These criteria are selected with regard to the intended application of this protocol and the limited availability of standard mixtures in this area. Corrective action should be taken with the column or PID before sample injections are made if the performance is deemed out-of-range. Under this regimen of blanks and system performance samples, approximately eight samples can be collected and analyzed in a three hour visit to each sampling location.

### 10.3 Method Precision and Accuracy

The purpose of the analytical approach outlined in this method is to provide presumptive information regarding the presence of selected VOCs and SVOCs emissions. In this context, precision and accuracy are to be determined. However, quality assurance criteria are described in Section 10.2 which insure the samples collected represent the indoor environment.

### 10.4 Range and Limits of Detection

The range and limits of detection of this method are highly compound dependent due to large differences in response of the portable GCs photoionization detector to the various

T014-B9

target compounds. Aromatic compounds and olefinic halogenated compounds will be detected at lower levels than the halomethanes or aliphatic hydrocarbons. The concentration range of application of this method is approximately two orders of magnitude.

T014-B10

TABLE B-1

ESTIMATED LIMITS OF DETECTION (LOD) FOR SELECTED VOCs  
BASED ON 1  $\mu$ L SAMPLE VOLUME

Compound	LOD (ng)	LOD (ppb)
Chloroform <sup>a</sup>	2	450
1,1,1-Trichloroethane <sup>a</sup>	2	450
Carbon tetrachloride <sup>a</sup>	2	450
Benzene	.006	2
1,2-Dichloroethane <sup>b</sup>	.05	14
Trichloroethylene <sup>b</sup>	.05	14
Tetrachloroethylene <sup>b</sup>	.05	14
1,2-Dibromoethane	.02	2
p-Xylene <sup>c</sup>	.02	4
m-Xylene <sup>c</sup>	.02	4
o-Xylene <sup>d</sup>	.01	3
Styrene <sup>d</sup>	.01	3

<sup>a</sup>Chloroform, 1,1,1-Trichloroethane, and Carbon tetrachloride coelute on 0.66 m 3% SP2100.

<sup>b</sup>1,2-Dichloroethane, Trichloroethylene, and Tetrachloroethylene coelute on 0.66 m 3% SP2100.

<sup>c</sup>p-Xylene and m-Xylene coelute on 0.66 m 3% SP2100.

<sup>d</sup>Styrene and o-Xylene coelute on 0.66 m 3% SP2100.

TABLE B-2

COMMERCIALY AVAILABLE  
PORTABLE VOC DETECTION INSTRUMENTS

Monitor	Detection principle	Range, ppm	Sensitivity	Response time, s	Accessories	Calibration Techniques	Weaknesses	Service Rate	Lack of Response	Cost, \$	Samp Rate L/m
550,551 555,580 (AID, Inc.)	PID, FID	0-200, 0-2000, 0-10,000	0.1 ppm at 0-200 ppm	<5		o Bag Sampling	o Umbilical cord too short o Digital readout hard to read o Flame out frequently	8 hrs		4,300	1.5
OVA 108, 128 Century Systems, Inc. (Foxboro)	FIU	0-10, 0-100, 0-1000, 0-10,000, 0-100,000	0.2 ppm (Model 128) 0.5 ppm (Model 108)	2  2	o Thermal Desorbers available o Optional GC available	o Hand Space o Direct Injection o Bag Samp.	o Battery failure o Sample line kinks o Compounds containing O <sub>2</sub> /N give low response o Neg. resp. to CO/CO <sub>2</sub>	8 hrs		6,300	
PI-101 (HMu Systems, Inc)	PID	1 1-20 1-200 1-2000	0.1 ppm Low molecular weights aromatics	<5	o Three lamps available o 9.5 (aromatics) o 10.2 (2-4 compounds) o 11.7 (halocarbons)	o External Gas Cyl. o Bag Samp.	o Three lamps - may miss something	10 hrs	o Cl hydrocarbons o CH <sub>4</sub>	4,955	0.5
TLV Sniffer (Bacharach)	Catalytic combustion	0-500 0-5000 0-50,000	2.0 ppm	5		o Bag Samp. o Head Space				900	
Ecoalyzer 400 (Energetics Science)	Catalytic combustion	0-100% LFL	1% LFL	15		o Bag Samp.	o Changes in gas temp/humidity affects response				
Hiran 1A (Foxboro)	IR	ppm to %	1 ppm	1,4,10 and 40						9,500	
Hiran 1B (Foxboro)	IR	ppm to %								12,500	
Scentor (Sentex)	GC/EC, Argon ionization PID		0.01 ppb Cl organics	2	Preconcentrator Thermal Desorption GC Columns Auto Cal. from Integral Gas Cylinder	o Internal gas cyl. o Preconcentrator o GC Column				12,950	
Photovac - Standard Automatic Computer Auto Comp. Communication	PID (UV Light)	0	0.1 ppb Benzene with signal-to-noise ratio 4:1. Good for aromatics	2	o Dual Column o Manual/Auto Injection o Column Cond. o Pre-flush o Auto Dial Mode o Programmable		o Column operates at ambient temp. o STU in lab, then to field at diff. temp o Can't inject liquid samp. o Light fractions interfere		o H <sub>2</sub> O o O <sub>2</sub>	6,995 8,995 10,500 10,955 12,955	
Photovac Tip	PID	0-2000 ppm	0.05 ppm Benzene	3							



T014-B13

TABLE B-4

SYSTEM PERFORMANCE CRITERIA FOR PORTABLE GC<sup>a</sup>

Criteria	Test Compound	Acceptable Range	Suggested Corrective Action
PID Response	Trichloroethylene	$\geq 10^8$ uV-sec/ng	Re-tune or replace lamp
Elution Time	Styrene	$2.65 \pm 0.15$ min	Inspect for leaks, adjust carrier flow
Resolution <sup>b</sup>	Benzene/Trichloroethylene	$\geq 1.4$	Replace column

<sup>a</sup>Based on analysis of a vapor mixture of benzene, styrene, and trichloroethylene.

<sup>b</sup>Define by:  $R + = 2d/(W_1+W_2)$ ; where d = distance between the peaks and W = peak width at base.

T014-B14

TABLE B-5

ESTIMATED LIMITS OF DETECTION (LOD) FOR SELECTED VOCs

Compound	LOD (ng)	LOD (ppb)
Chloroform <sup>a</sup>	2	450
1,1,1-Trichloroethane <sup>a</sup>	2	450
Carbon tetrachloride <sup>a</sup>	2	450
Benzene	.006	2
1,2-Dichloroethane <sup>b</sup>	.05	14
Trichloroethylene <sup>b</sup>	.05	14
Tetrachloroethylene <sup>b</sup>	.05	14
1,2-Dibromoethane	.02	2
p-Xylene <sup>c</sup>	.02	4
m-Xylene <sup>c</sup>	.02	4
o-Xylene <sup>d</sup>	.01	3
Styrene <sup>d</sup>	.01	3

<sup>a</sup>Chloroform, 1,1,1-Trichloroethane, and Carbon tetrachloride coelute on 0.66 m 3% SP2100.

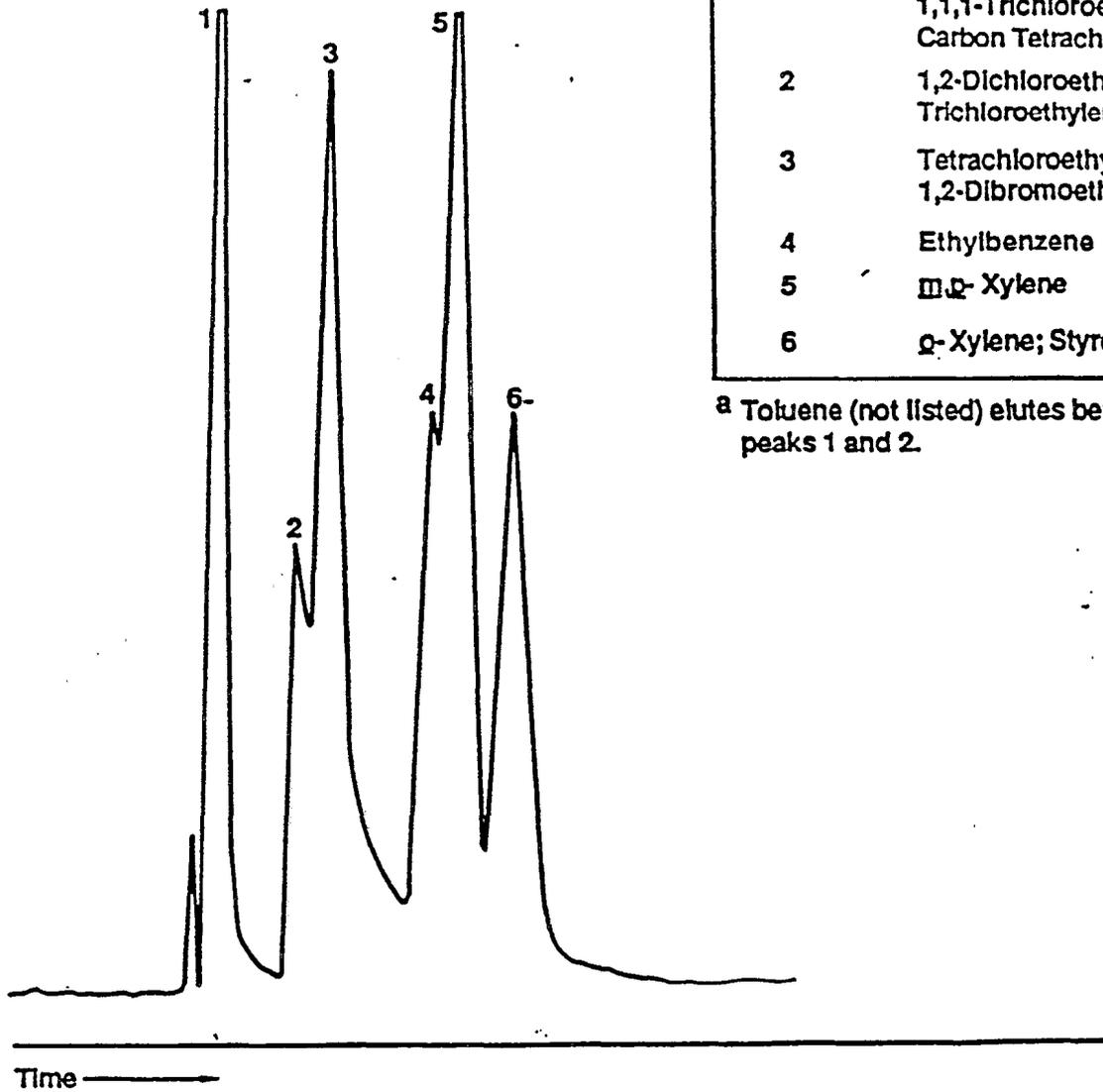
<sup>b</sup>1,2-Dichloroethane, Trichloroethylene, and Tetrachloroethylene coelute on 0.66 m 3% SP2100.

<sup>c</sup>p-Xylene and m-Xylene coelute on 0.66 m 3% SP2100.

<sup>d</sup>Styrene and o-Xylene coelute on 0.66 m 3% SP2100.

## Peak Assignments For Standard Mixture

Peak No.	Compound(s) <sup>a</sup>
1	Benzene; Chloroform; 1,1,1-Trichloroethane; Carbon Tetrachloride
2	1,2-Dichloroethane; Trichloroethylene
3	Tetrachloroethylene; 1,2-Dibromoethane
4	Ethylbenzene
5	<i>m,p</i> -Xylene
6	<i>o</i> -Xylene; Styrene



<sup>a</sup> Toluene (not listed) elutes between peaks 1 and 2.

FIGURE B-1. TYPICAL CHROMATOGRAM OF VOCs DETERMINED BY A PORTABLE GC

## APPENDIX C

### INSTALLATION AND OPERATION PROCEDURES FOR U.S. ENVIRONMENTAL PROTECTION AGENCY'S URBAN AIR TOXIC POLLUTANT PROGRAM SAMPLER

#### 1. Scope

- 1.1 The subatmospheric sampling system described in this method has been modified and redesigned specifically for use in USEPA's Urban Air Toxic Pollutant Program (UATP), a joint project of USEPA's Office of Air Quality Planning and Standards, the Environmental Monitoring Systems Laboratory, and the participating state air pollution control agencies. The purpose of UATP is to provide analytical support to the states in their assessment of potential health risks from certain toxic organic compounds that may be present in urban atmospheres. The sampler is described in the paper, "Automatic Sampler for Collection of 24-Hour Integrated Whole-Air Samples for Organic Analysis," to be presented at the 1988 Annual Meeting of APCA, Dallas, Texas, June, 1988 (Paper No. 88-150.3).
- 1.2 The sampler is based on the collection of whole air samples in 6-liter, SUMMA® passivated stainless steel canisters. The sampler features electronic timer for ease, accuracy and flexibility of sample period programming, an independently settable presample warm-up and ambient air purge period, protection from loss of sample due to power interruptions, and a self-contained configuration housed in an all-metal portable case, as illustrated in Figure C-1.
- 1.3 The design of the sampler is pumpless, using an evacuated canister to draw the ambient sample air into itself at a fixed flow rate (3-5 cm<sup>3</sup>/min) controlled by an electronic mass flow controller. Because of the relatively low sample flow rates necessary for the integration periods, auxiliary flushing of the sample inlet line is provided by a small, general-purpose vacuum pump (not in contact with the sample air stream). Further, experience has shown that inlet lines and surfaces sometimes build up or accumulate substantial concentrations of organic materials under stagnant (zero flow rate) conditions. Therefore such lines and surfaces need to be purged and equilibrated to the sample air for some time prior to the beginning of the actual sample collection period. For this reason, the sampler includes dual timers, one of which is set to start the pump several hours prior to the specified start of the sample period to purge the inlet lines and

surfaces. As illustrated in Figure C-1, sample air drawn into the canister passes through only four components: the heated inlet line, a 2-micron particulate filter, the electron flow controller, and the latching solenoid valve.

## 2. Summary of Method

- 2.1 In operation, timer 1 is set to start the pump about 6 hours before the scheduled sample period. The pump draws sample air in through the sample inlet and particulate filter to purge and equilibrate these components, at a flow rate limited by the capillary to approximately  $100 \text{ cm}^3/\text{min}$ . Timer 1 also energizes the heated inlet line to allow it to come up to its controlled temperature of 65 to 70 degrees C, and turns on the flow controller to allow it to stabilize. The pump draws additional sample air through the flow controller by way of the normally open port of the 3-way solenoid valve. This flow purges the flow controller and allows it to achieve a stable controlled flow at the specified sample flow rate prior to the sample period.
- 2.2 At the scheduled start of the sample period, timer 2 is set to activate both solenoid valves. When activated, the 3-way solenoid valve closes its normally open port to stop the flow controller purge flow and opens its normally closed port to start flow through the aldehyde sample cartridges. Simultaneously, the latching solenoid valve opens to start sample flow into the canister.
- 2.3 At the end of the sample period, timer 2 closes the latching solenoid valve to stop the sample flow and seal the sample in the canister and also de-energizes the pump, flow controller, 3-way solenoid, and heated inlet line. During operation, the pump and sampler are located external to the sampler. The 2.4 meter (8 foot) heated inlet line is installed through the outside wall, with most of its length outside and terminated externally with an inverted glass funnel to exclude precipitation. The indoor end is terminated in a stainless steel cross fitting to provide connections for the canister sample and the two optional formaldehyde cartridge samples.

## 3. Sampler Installation

- 3.1 The sampler must be operated indoors with the temperature between  $20$  to  $32^\circ\text{C}$  ( $68$  to  $90^\circ\text{F}$ ). The sampler case should be located conveniently

on a table, shelf, or other flat surface. Access to a source of 115 vac line power (500 watts min) is also required. The pump is removed from the sampler case and located remotely from the sampler (connected with a 1/4 inch O.D. extension tubing and a suitable electrical extension cord).

### 3.2 Electrical Connections (Figure C-1)

3.2.1 The sampler cover is removed. The sampler is not plugged into the 115 vac power until all other electrical connections are completed.

3.2.2 The pump is plugged into its power connector (if not already connected) and the battery connectors are snapped onto the battery packs on the covers of both timers.

3.2.3 The sampler power plug is inserted into a 115 volts ac line grounded receptacle. The sampler must be grounded for operator safety. The electrical wires are routed and tied so they remain out of the way.

### 3.3 Pneumatic Connections

3.3.1 The length of 1/16 inch O.D. stainless steel tubing is connected from port A of the sampler (on the right side of the flow controller module) to the air inlet line.

3.3.2 The pump is connected to the sampler with 1/4 inch O.D. plastic tubing. This tubing may be up to 7 meters (20 feet) long. A short length of tubing is installed to reduce pump noise. All tubing is conveniently routed and, if necessary, tied in place.

## 4. Sampler Preparation

### 4.1 Canister

4.1.1 The sample canister is installed no more than 2 days before the scheduled sampling day.

4.1.2 With timer #1 ON, the flow controller is allowed to warm up for at least 15 minutes, longer if possible.

4.1.3 An evacuated canister is connected to one of the short lengths of 1/8 inch O.D. stainless steel tubing from port B (solenoid valve) of the sampler. The canister valve is left closed. The Swagelok fitting on the canister must not be cross-threaded. The connection is tightened snugly with a wrench.

- 4.1.4 The end of the other length of stainless steel tubing from port B (solenoid valve) is connected with a Swagelok plug.
- 4.1.5 If duplicate canisters are to be sampled, the plug is removed from the second 1/8 inch O.D. stainless steel tubing from port B (solenoid valve) and the second canister is connected. The canister valve is left closed.
- 4.1.6 The ON button of timer #2 is pressed. The flow through the flow controller should be stopped by this action.
- 4.1.7 The flow controller switch is turned to "READ" and the zero flow reading is obtained. If this reading is not stable, wait until the reading is stabilized.
- 4.1.8 The flow controller switch is turned to "SET" and the flow setting is adjusted to the algebraic SUM of the most recent entry on Table C-1 and the zero reading obtained in step 4.1.7 (If the zero reading is negative, SUBTRACT the zero reading from the Table C-1 value). Be sure to use the correct Table C-1 flow value for one or two canisters, as appropriate. [Note: If the analytical laboratory determines that the canister sample pressure is too low or too high, a new flow setting or settings will be issued for the sampler. The new flow setting should be recorded in Table C-1 and used until superseded by new settings.]
- 4.1.9 Timer #2 is turned OFF to again start the flow through the flow controller. With the pump (timer #1) ON and the sampling valve (timer #2) OFF, the flow controller is turned to "READ" and the flow is verified to be the same as the flow setting made in step 4.1.8. If not, the flow setting is rechecked in step 4.1.8 and the flow setting is readjusted if necessary.
- 4.1.10 The OFF button of timer #1 is pressed to stop the pump.
- 4.1.11 The canister valve(s) are fully opened.

## 4.2 Timers

- 4.2.1 Timer #2 is set to turn ON at the scheduled ON time for the sample period, and OFF at the scheduled OFF time. (See the subsequent section on setting the timers.)

Normal ON time: 12:00 AM on the scheduled sampling day.

11:50 PM on the scheduled sampling day.

(The OFF time is 11:59 PM instead of 12:00 AM so that the day number for the OFF time is the same as the day number for the ON time.) Be sure to set the correct day number.

4.2.2 Timer #1 is set to turn ON six (6) hours before the beginning of the scheduled sample period and OFF at the scheduled OFF time for the sample period (same OFF time as for timer #2). (See the subsequent section on setting the timers.) Normal ON time: 06:00 PM on the day prior to the scheduled sampling day. Normal OFF time: 11:59 PM on the scheduled sampling day. [Note: The timers are wired so that the pump will be on whenever either timer is on. Thus the pump will run if timer #2 is ON even if timer #1 is OFF.]

4.2.3 The elapsed time meter is set to 0.

#### 4.3 Sampler Check

4.3.1 The following must be verified before leaving the sampling site:

- (1) Canister(s) is (are) connected properly and the unused connection is capped if only one canister is used.
- (2) Canister valve(s) is (are) opened.
- (3) Both timers are programmed correctly for the scheduled sample period.
- (4) Both timers are set to "AUTO".
- (5) Both timers are initially OFF.
- (6) Both timers are set to the correct current time of day and day number.
- (7) Elapsed time meter is set to 0.

#### 4.4 Sampler Recovery (Post Sampling)

4.4.1 The valve on the canister is closed.

4.4.2 The canister is disconnected from the sampler, the sample data sheet is completed, and the canister is prepared for shipment to the analytical laboratory.

4.4.3 If two canisters were sampled, step 2.4.2 is repeated for the other canister.

#### 5. Timer Setting

Since the timers are 7-day timers, the days of the week are numbered from 1 to 7. The assignment of day numbers to days of the week is

indicated on the timer keypad: 1 = Sunday, 2 = Monday, 3 = Tuesday, 4 = Wednesday, 5 = Thursday, 6 = Friday, and 7 = Saturday. This programming is quite simple, but some timers may malfunction or operate erratically if not programmed exactly right. To assure correct operation, the timers should be reset and completely reprogrammed "from scratch" for each sample. The correct current time of day is re-entered to reprogram the timer. Any program in the timer's memory is erased by resetting the timer (pressing the reset button). The timer is set by the following:

- (1) pressing the reset button,
- (2) entering the correct day number and time of day,
- (3) entering the ON and OFF times for the sample period, and
- (4) verifying that the ON and OFF time settings are correct.

#### 5.1 Timer Reset

The timer reset button is pressed, which is recessed in a small hole located just above the LED (light emitting diode) indicator light. A small object that will fit through the hole, such as a pencil, match, or pen is used to press the timer. After reset, the timer display should show [1] [10:00]. [Note: The timers may operate erratically when the batteries are discharged, which happens when the sampler is unplugged or without power for several hours. When the sampler is again powered up, several hours may be required to recharge the batteries. To avoid discharging the batteries, the battery pack should be disconnected from the timer when the sampler is unplugged.]

#### 5.2 Date and Time Entry

The selector switch is turned to SET and the number button corresponding to the day number is pressed. (For example, a "2" is pressed for Monday.) The current time of day is entered. (For example, if the time is 9:00 AM, 900 is pressed.) AM or PM is pressed as applicable. (Display should show [2] ['9:00] for 9:00 AM Monday.) [Note: ' indicates AM and indicates PM.] The CLOCK button is pressed. (Display should show [-] [--:--]) If an error is made, [E] [EE:EE] is shown on the display. The CLEAR button is pressed and the above steps are repeated. The selector switch is turned to AUTO or MAN to verify correct time setting.

5.3 ON and OFF Entry

The selector switch is turned to SET. The ON and OFF program is entered in the following order: day, number, time, AM or PM, ON or OFF. (Example: To turn ON at 12:00 AM on day 5 (Thursday); 5, 1200, AM, ON is entered). (Example: To turn OFF at 11:59 PM on day 5 (Thursday), 5, 11:59, PM, OFF is entered.) If the display indicates an error (|E| |EE:EE|), the timer is reset. The selector switch is turned to AUTO.

5.4 ON and OFF Verification

5.4.1 The selector switch is turned to REVIEW. The number of the scheduled sample day is pressed. ON is pressed. The display should show the time of the beginning of the sample period (for example, |5| |'12:00|). [' indicates AM.] ON is pressed again. The display should show |5| |--:--|, indicating no other ON times are programmed.

5.4.2 OFF is pressed. The display should show the time of the end of the sample period, (for example, |5| |, 11:59|). PM is indicated by the "," mark before the time. OFF is pressed again. The display should show |5| |--:--|, indicating no other OFF times are programmed. The selector is switched to AUTO. If anything is incorrect, the timer is reset and reprogrammed.

TABLE C-1

NET FLOW CONTROLLER SETTING

<u>DATE</u>	<u>1 CANISTER</u>	<u>2 CANISTERS</u>
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

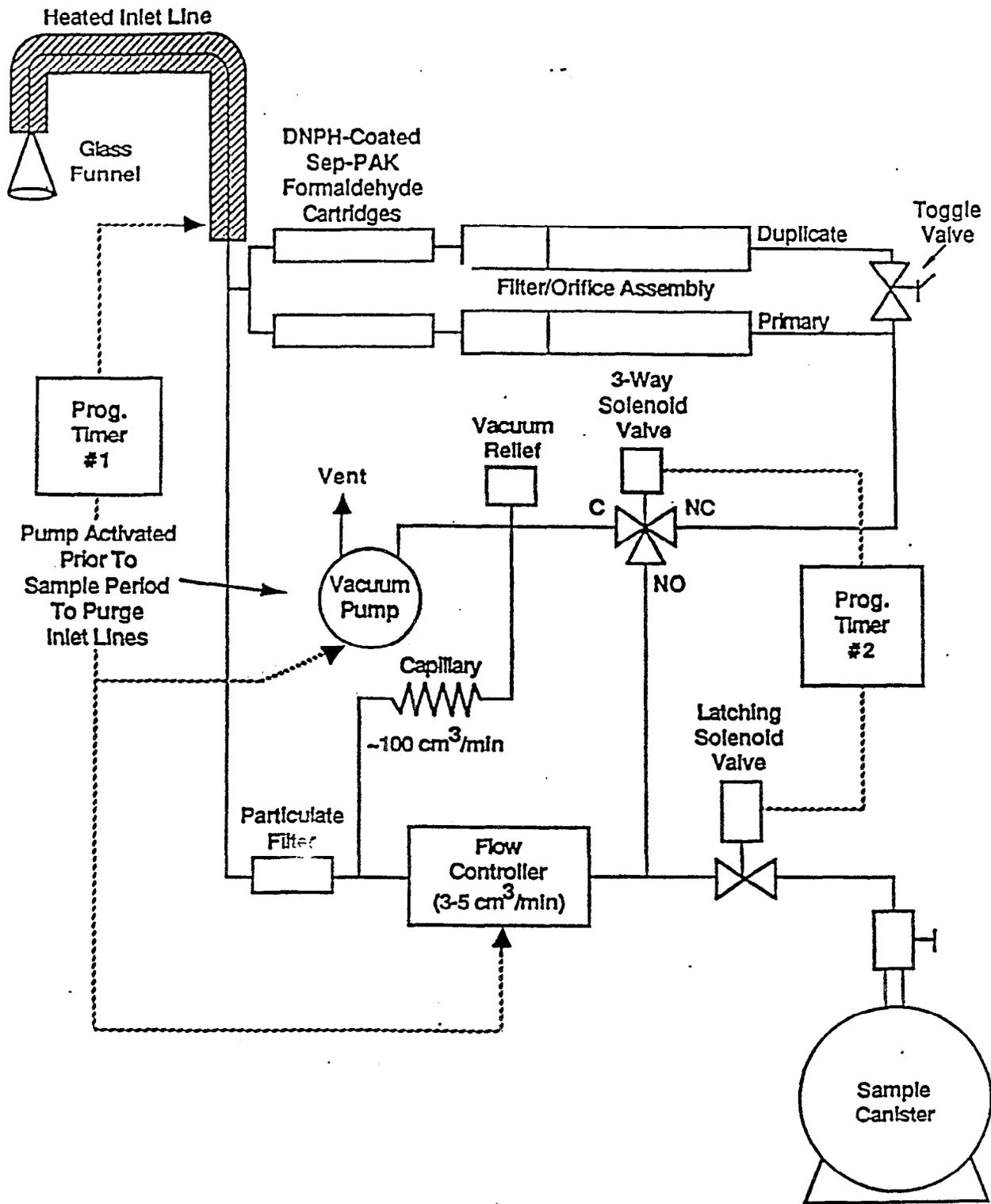


FIGURE C-1. U.S. ENVIRONMENTAL PROTECTION AGENCY UATP SAMPLER SCHEMATIC OF SAMPLE INLET CONNECTIONS

**APPENDIX C**  
**CANISTER SAMPLING DATA SHEETS**

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**CANISTER SAMPLING DATA SHEET**

Site: \_\_\_\_\_

Performed by: \_\_\_\_\_

Date	Canister Number	Sampling Period (hour)		Sampling Time (min)	Initial* Canister Vacuum (in/Hg)	Final* Canister Vacuum/ Pressure (in/Hg PSI)	Initial Flow (cc/min)	Final Flow (cc/min)	Comments
		Start	Stop						

Data Checked by: \_\_\_\_\_

Date: \_\_\_\_\_

\*Initial and Final Canister Vacuum to be provided by the laboratory.

