



# Standard Test Method for Determination of Volatile Organic Chemicals in Atmospheres (Canister Sampling Methodology)<sup>1</sup>

This standard is issued under the fixed designation D 5466; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method describes a procedure for sampling and analysis of volatile organic compounds (VOCs) in ambient, indoor, or workplace atmospheres. The test method is based on the collection of air samples in passivated stainless steel canisters. The VOCs are subsequently removed from the canisters, separated by gas chromatography, and measured by a mass spectrometric detector. This test method describes procedures for sampling into canisters to final pressures both above and below atmospheric pressure (respectively referred to as pressurized and subatmospheric pressure sampling).<sup>2</sup>

1.2 This test method is applicable to specific VOCs that have been tested and determined to be stable when stored in pressurized and subatmospheric pressure canisters. Numerous compounds, many of which are chlorinated VOCs, have been successfully tested for storage stability in pressurized canisters (1, 2).<sup>3</sup> While not as extensive, documentation is currently also available demonstrating stability of VOCs in subatmospheric pressure canisters.

1.3 The organic compounds that have been successfully collected in pressurized canisters by this test method are listed in Table 1. These compounds have been successfully measured at the parts per billion by volume (ppbv) level. This test method is applicable to concentrations of VOC from the detection limit to 300 ppb by volume. Above this concentration samples require dilution with dry ultra high purity nitrogen or air.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Safety practices should be part of the user's SOP manual.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D-22 on Sampling and Analysis of Atmospheres and is the direct responsibility of Subcommittee D22.05 on Indoor Air.

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<sup>2</sup> This test method is based on EPA Compendium Method TO-14, "The Determination of Volatile Organic Compounds (VOCs) in Ambient Air Using SUMMA Passivated Canister Sampling and Gas Chromatographic Analysis," May 1988.

<sup>3</sup> The **boldface** numbers in parentheses refer to the list of references at the end of the standard.

## 2. Referenced Documents

### 2.1 ASTM Standards:

D 1356 Terminology Relating to Sampling and Analysis of Atmospheres<sup>4</sup>

D 1357 Practice for Planning and Sampling of the Ambient Atmosphere<sup>4</sup>

E 260 Practice for Packed Column Gas Chromatography<sup>5</sup>

E 355 Practice for Gas Chromatography Terms and Relationships<sup>5</sup>

### 2.2 Other Documents:

U.S. Environmental Protection Agency Technical Assistance Document for Sampling and Analysis of Toxic Organic Compounds in Ambient Air (3)

Laboratory and Ambient Air Studies (4-20)

## 3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D 1356. Other pertinent abbreviations and symbols are defined within this practice at point of use.

### 3.2 Definitions of Terms Specific to This Standard:

3.2.1 *absolute canister pressure*— $P_g + P_a$ , where  $P_g$  = gage pressure in the canister. (KPa, psi) and  $P_a$  = barometric pressure (see 5.2).

3.2.2 *absolute pressure*—pressure measured with reference to absolute zero pressure (as opposed to atmospheric pressure), usually expressed as kPa, mm Hg, or psia.

3.2.3 *certification*—the process of demonstrating with humid zero air and humid calibration gases that the sampling systems components and the canister will not change the concentrations of sampled and stored atmospheres.

3.2.4 *cryogen*—a refrigerant used to obtain very low temperatures in the cryogenic trap of the analytical system. A typical cryogen is liquid argon (bp  $-185.7^\circ\text{C}$ ) or liquid nitrogen (bp  $-195^\circ\text{C}$ ).

3.2.5 *dynamic calibration*—calibration of an analytical system using calibration gas standard concentrations generated by diluting known concentration compressed gas standards with purified, humidified inert gas.

3.2.5.1 *Discussion*—Such standards are in a form identical or very similar to the samples to be analyzed. Calibration

<sup>4</sup> Annual Book of ASTM Standards, Vol 11.03.

<sup>5</sup> Annual Book of ASTM Standards, Vol 14.02.

**TABLE 1 Volatile Organic Compounds Known to Have Been Analyzed by the Canister Method**

Compound (Synonym)	Formula	Molecular Weight	Boiling Point (°C)	Melting Point (°C)	Cas Number
Freon 12 (Dichlorodifluoromethane)	Cl <sub>2</sub> CF <sub>2</sub>	120.91	-29.8	-158.0	
Methyl chloride (Chloromethane)	CH <sub>3</sub> Cl	50.49	-24.2	-97.1	74-87-3
Freon 114 (1,2-Dichloro-1,1,2,2-tetrafluoroethane)	C <sub>2</sub> Cl <sub>2</sub> CF <sub>2</sub>	170.93	4.1	-94.0	
Vinyl chloride (Chloroethylene)	CH <sub>2</sub> =CHCl	62.50	-13.4	-1538.0	75-01-4
Methyl bromide (Bromomethane)	CH <sub>3</sub> Br	94.94	3.6	-93.6	74-83-9
Ethyl chloride (Chloroethane)	CH <sub>3</sub> CH <sub>2</sub> Cl	64.52	12.3	-136.4	75-00-3
Freon 11 (Trichlorofluoromethane)	CCl <sub>3</sub> F	137.38	23.7	-111.0	
Vinylidene chloride (1,1-Dichloroethene)	C <sub>2</sub> H <sub>2</sub> Cl <sub>2</sub>	96.95	31.7	-122.5	75-35-4
Dichloromethane (Methylene chloride)	CH <sub>2</sub> Cl <sub>2</sub>	84.94	39.8	-95.1	75-09-2
Freon 113 (1,1,2-Trichloro-1,2,2-trifluoroethane)	CF <sub>2</sub> ClCCl <sub>2</sub> F	187.38	47.7	-36.4	
1,1-Dichloroethane (Ethylidene chloride)	CH <sub>3</sub> CHCl <sub>2</sub>	98.96	57.3	-97.0	74-34-3
cis-1,2-Dichloroethylene	CHCl=CHCl	96.94	60.3	-80.5	
Chloroform (Trichloromethane)	CHCl <sub>3</sub>	119.38	61.7	-63.5	67-66-3
1,2-Dichloropropane (Ethylene dichloride)	C <sub>2</sub> H <sub>4</sub> Cl <sub>2</sub>	98.96	83.5	-35.3	107-06-2
Methyl chloroform (1,1,1-Trichloroethane)	CH <sub>3</sub> CCl <sub>3</sub>	133.41	74.1	-30.4	71-55-6
Benzene (Cyclohexatriene)	C <sub>6</sub> H <sub>6</sub>	78.12	80.1	5.5	71-43-2
Carbon tetrachloride (Tetrachloromethane)	CCl <sub>4</sub>	153.82	76.5	-23.0	56-23-5
1,2-Dichloropropane (Propylene dichloride)	CH <sub>3</sub> CHClCH <sub>2</sub> Cl	112.99	96.4	-100.4	78-87-5
Trichloroethylene (Trichloroethene)	C <sub>2</sub> HCl=CCl <sub>2</sub>	131.29	87	-73.0	79-01-6
cis-1,3-Dichloropropene (cis-1,3-dichloropropylene)	CH <sub>3</sub> CC=CHCl	110.97	76		
trans-1,3-Dichloropropene (cis-1,3-Dichloropropylene)	C <sub>2</sub> H <sub>3</sub> Cl <sub>2</sub>	110.97	112.0		
1,1,2-Trichloroethane (Vinyl trichloride)	CH <sub>2</sub> ClCHCl <sub>2</sub>	133.41	113.8	-36.5	79-00-5
Toluene (Methyl benzene)	C <sub>6</sub> H <sub>5</sub> CH <sub>3</sub>	92.15	110.6	-95.0	108-88-3
1,2-Dibromoethane (Ethylene dibromide)	BrCH <sub>2</sub> CH <sub>2</sub> Br	187.88	131.3	9.8	106-93-4
Tetrachloroethylene (Perchloroethylene)	Cl <sub>2</sub> C=CCl <sub>2</sub>	165.83	121.1	-19.0	127-18-4
Chlorobenzene (Phenyl chloride)	C <sub>6</sub> H <sub>5</sub> Cl	112.56	132.0	-45.6	108-90-7
Ethylbenzene	C <sub>6</sub> H <sub>5</sub> C <sub>2</sub> H <sub>5</sub>	106.17	136.2	-95.0	100-41-4
m-Xylene (1,3-Dimethylbenzene)	1,3-(CH <sub>3</sub> ) <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	106.17	139.1	-47.9	
p-Xylene (1,4-Dimethylxylene)	1,4-(CH <sub>3</sub> ) <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	106.17	138.3	13.3	
Styrene (Vinyl benzene)	C <sub>6</sub> H <sub>5</sub> CH=CH <sub>2</sub>	104.16	145.2	-30.6	100-42-5
1,1,2,2-Tetrachloroethane	CHCl <sub>2</sub> CHCl <sub>2</sub>	167.85	146.2	-36.0	79-34-5
o-Xylene (1,2-Dimethylbenzene)	1,2-(CH <sub>3</sub> ) <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	106.17	144.4	-25.2	
1,3,5-Trimethylbenzene (Mesitylene)	1,3,5-(CH <sub>3</sub> ) <sub>3</sub> C <sub>6</sub> H <sub>6</sub>	120.20	164.7	-44.7	108-67-8
1,2,4-Trimethylbenzene (Pseudocumene)	1,2,4-(CH <sub>3</sub> ) <sub>3</sub> C <sub>6</sub> H <sub>6</sub>	120.20	169.3	-43.8	95-63-6
m-Dichlorobenzene (1,3-Dichlorobenzene)	1,3-Cl <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	147.01	173.0	-24.7	541-73-1
Benzyl chloride (α-Chlorotoluene)	C <sub>6</sub> H <sub>5</sub> CH <sub>2</sub> Cl	126.59	179.3	-39.0	100-44-7
o-Dichlorobenzene (1,2-Dichlorobenzene)	1,2-Cl <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	147.01	180.5	-17.0	95-50-1
p-Dichlorobenzene (1,4-Dichlorobenzene)	1,4-Cl <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	147.01	174.0	53.1	106-46-7
1,2,4-Trichlorobenzene Hexachlorobutadiene (1,1,2,3,4,4-Hexachloro-1,3-butadiene)	1,2,4-Cl <sub>3</sub> C <sub>6</sub> H <sub>3</sub>	181.45	213.5	17.0	120-82-1

ASTM D5466-95

<https://standards.iteh.ai/catalog/standards/sist/318f8576-d078-4ea4-a35e-b2f2bca35f2a/astm-d5466-95>

standards are introduced into the inlet of the sampling or analytical system in the same manner as authentic field samples.

3.2.6 *gage pressure*—pressure measured above ambient atmospheric pressure (as opposed to absolute pressure). Zero gage pressure is equal to ambient atmospheric (barometric) pressure.

3.2.7 *megabore column*—chromatographic column having an internal diameter (I.D.) greater than 0.50 mm.

3.2.7.1 *Discussion*—The Megabore column is a trademark of the J & W Scientific Co. For purposes of this test method, Megabore refers to chromatographic columns with 0.53 mm I.D.

3.2.8 *MS-SCAN*—the GC is coupled to a Mass Spectrometer (MS) programmed to scan all ions over a preset range repeatedly during the GC run.

3.2.8.1 *Discussion*—As used in the current context, this procedure serves as a qualitative identification and characterization of the sample.

3.2.9 *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.

3.2.10 *pressurized sampling*—collection of an air sample in a canister with a (final) canister pressure above atmospheric pressure, using a sample pump.

3.2.11 *qualitative accuracy*—the ability of an analytical system to correctly identify compounds.

3.2.12 *quantitative accuracy*—the ability of an analytical system to correctly measure the concentration of an identified compound.

3.2.13 *static calibration*—calibration of an analytical system using standards in a form different than the samples to be analyzed.

3.2.13.1 *Discussion*—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.

3.2.14 *subatmospheric sampling*—collection of an air sample in an evacuated canister to a (final) canister pressure below atmospheric pressure, with or without the assistance of a sampling pump.

3.2.14.1 *Discussion*—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.

#### 4. Summary of Test Method

4.1 Both subatmospheric pressure and pressurized sampling modes use an evacuated canister. A sampling line less than 2 % of the volume of the canister or a pump-ventilated sample line are used during sample collection. Pressurized sampling requires an additional pump to provide positive pressure to the sample canister. A sample of air is drawn through a sampling train comprising components that regulate the rate and duration of sampling into a precleaned and pre-evacuated SUMMA® passivated canister.

**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 87/31	5.01
Methyl chloride (Chloromethane)	50/100 52/34	5.69
Freon 114 (1,2-Dichloro-1,1,2,2-tetrafluoroethane)	85/100 135/56	6.55
Vinyl chloride (Chloroethene)	87/33 62/100 27/125	6.71
Methyl bromide (Bromomethane)	64/32 94/100 96/85	7.83
Ethyl chloride (Chloroethane)	64/100 29/140 27/140	8.43
Freon 11 (Trichlorofluoromethane)	101/100 103/67	9.97
Vinylidene chloride (1,1-Dichloroethylene)	61/100 96/55 63/31	10.93
Dichloromethane (Methylene chloride)	49/100 84/65 86/45	11.21
Freon 113 (1,1,2-Trichloro-1,2,2-trifluoroethane)	151/100 101/140 103/90	11.60
1,1-Dichloroethane (Ethylidene dichloride)	63/100 27/64 65/33	12.50
cis-1,2-Dichloroethylene	61/100 96/60 98/44	13.40
Chloroform (Trichloromethane)	83/100 85/65 47/35	13.75
1,2-Dichloroethane (Ethylene dichloride)	62/100 27/70 64/31	14.39
Methyl chloroform (1,1,1-Trichloroethane)	97/100 99/64 61/61	14.62
Benzene (Cyclohexatriene)	78/100 77/25 50/35	15.04
Carbon tetrachloride (Tetrachloromethane)	117/100 119/97	15.18
1,2-Dichloropropane (Propylene dichloride)	63/100 41/90	15.83

**TABLE 2 Continued**

Compound	Ion/ Abundance (amu/% base peak)	Expected Retention Time (min)
Trichloroethylene (Trichloroethane)	62/70 130/100 132/92	16.10
cis-1,3-Dichloropropene	95/87 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
Benzyl chloride (α-Chlorotoluene)	91/100 126/26	23.32
p-Dichlorobenzene (1,4-Dichlorobenzene)	146/100 148/65 111/40	23.41
o-Dichlorobenzene (1,2-Dichlorobenzene)	146/100 148/65 111/40	23.88
1,2,4-Trichlorobenzene	180/100 182/98 184/30	26.71
Hexachlorobutadiene (1,1,2,3,4,4-Hexachloro-1,3-butadiene)	225/100 227/66 223/60	27.68

4.2 After the air sample is collected, the canister isolation valve is closed, the canister is removed from the sampler, an identification tag is attached to the canister, and the canister is transported to a laboratory for analysis.

4.3 Upon receipt at the laboratory, the data on the canister tag are recorded and the canister is attached to a pressure gage which will allow accurate measurement of the final canister pressure. During analysis, water vapor may be reduced in the gas stream by a Nafion 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 a mass spectrometer. Compound identification and quantitation are performed with this test method.

4.4 A mass spectrometric detector (MS coupled to a GC) is the principal analytical tool used for qualitative and quantitative analysis because it allows positive compound identification. MS detectors include, but are not limited to, magnetic sector mass analyzers, quadrupole mass filters, combined magnetic sector-electrostatic sector mass analyzers, time-of-flight mass analyzers and ion trap mass spectrometers.

#### 4.4.1 Comparison of GC/MS–Full Scan and GC/MS–SIM:

##### 4.4.1.1 GC/MS–Full Scan:

- (1) Positive nontarget compound identification possible,
- (2) Less sensitivity than GC/MS–SIM,
- (3) Greater sample volume may be required compared to SIM,
- (4) Resolve co-eluting interfering ions is possible,
- (5) Positive compound identification,
- (6) Quantitative determination of compounds on calibration list, and
- (7) Qualitative and semiquantitative determination of compounds not contained on calibration list.

##### 4.4.1.2 GC/MS–SIM:

- (1) Can't identify non-target compounds,
- (2) Less operator interpretation, and
- (3) Higher sensitivity than GC/MS–full scan.

4.4.2 The GC/MS–full 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. As the number of ions monitored simultaneously in a GC/MS–SIM analysis increases, the sensitivity of this technique approaches GC/MS–SCAN. The practical limit for GC/MS–SIM is reached at about 4 to 5 ions monitored simultaneously.

## 5. Significance and Use

5.1 VOCs are emitted into the ambient, indoor, and workplace atmosphere from a variety of sources. In addition to the emissions from the use of various products, appliances, and building materials, fugitive or direct emissions from ambient sources such as manufacturing processes further complicate air composition. Many of these VOC compounds are acute or chronic toxins. Therefore, their determination in air is necessary to assess human health impacts.

5.2 The use of canisters is particularly well suited for the collection and analysis of very volatile, stable compounds in atmosphere (for example, vinyl chloride). This test method collects and analyzes whole gas samples and is not subject to high volatility limitations.

5.3 VOCs can be successfully collected in passivated stainless steel canisters. Collection of atmospheric samples in canisters provides for: (1) convenient integration of air samples

over a specific time period (for example, 8 to 24 h), (2) remote sampling and central laboratory analysis, (3) ease of storing and shipping samples, (4) unattended sample collection, (5) analysis of samples from multiple sites with one analytical system, (6) dilution or additional sample concentration to keep the sample size introduced into the analytical instrument within the calibration range, (7) collection of sufficient sample volume to allow assessment of measurement precision or analysis, or both, of samples by several analytical systems, and (8) can be performed in remote access areas using a vacuum regulator flow controller if electricity is not available.

5.4 Interior surfaces of the canisters are treated by the SUMMA® passivation process,<sup>6</sup> in which a pure chromium oxide is formed on the surface.

5.5 This test method can be applied to sampling and analysis of compounds that can be quantitatively recovered from the canisters. The typical range of VOC applicable to this test method are ones having saturated vapor pressures at 25°C greater than 15 Pa (10<sup>-1</sup> mm Hg).

5.6 Recovery and stability studies must be conducted on any compound not listed in Table 1 before expanding the use of this test method to additional compounds.

## 6. Interferences and Limitations

6.1 For those applications where a membrane dryer (for example, Nafion) is used, interferences can occur in sample analysis if moisture accumulates in the dryer (see 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.

NOTE 1—Removing moisture from samples is not necessary with GC/MS systems that are differentially pumped and which do not employ membrane drying apparatus.

6.2 Contamination may occur in the sampling system if canisters are not properly cleaned before use. Additionally, all other sampling equipment (for example, pump and flow controllers) must 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 11.1 and 11.2, respectively. In addition, sufficient system and field blank samples shall be analyzed to detect contamination as soon as it occurs.

6.3 If the GC/MS analytical system employs a Nafion permeable membrane dryer or equivalent to remove water vapor selectively from the sample stream, polar organic compounds will permeate this membrane concurrently with the moisture. Consequently, the analyst must calibrate his or her system with the specific organic constituents under examination. For quantitative analysis of polar compounds analytical systems may not employ Nafion permeable membrane dryers.

## 7. Apparatus

7.1 Subatmospheric pressure and pressurized canister sampling systems are commercially available and have been

<sup>6</sup> SUMMA® process is a registered trademark of Moleetrics Inc., 4000 East 89th St., Cleveland, OH 44105.

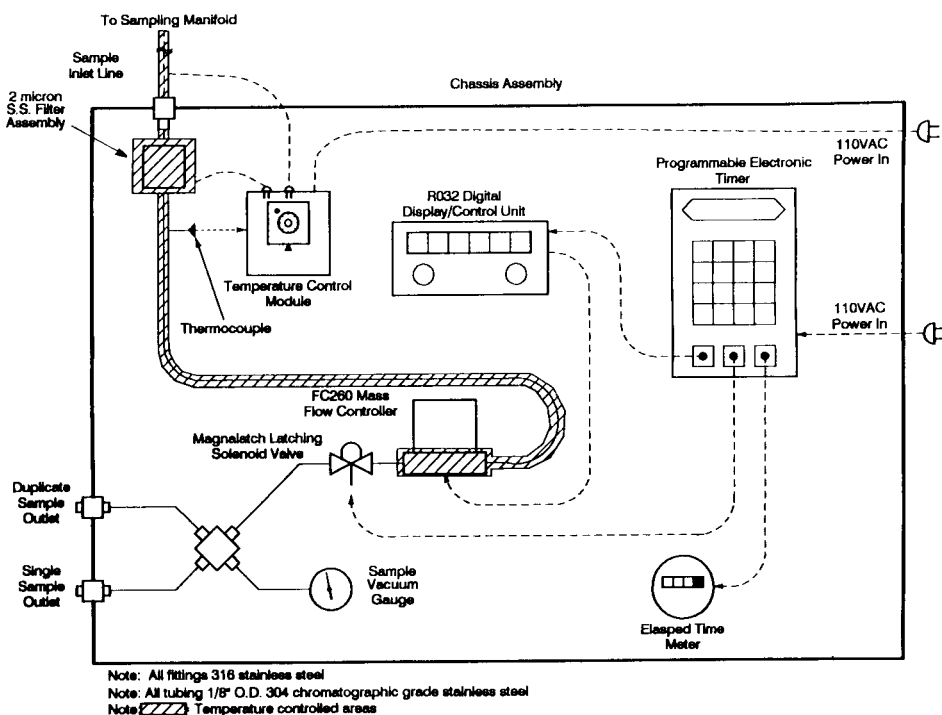


FIG. 1 Sampler Configuration For Subatmospheric Pressure Canister Sampling

certified for VOC testing in air (20). Several configurations of standard hardware can be used successfully as canister sampling units.

7.1.1 Subatmospheric Pressure (see Fig. 1).

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

7.1.1.2 Canister, Leak-free stainless steel pressure vessels of desired volume (for example, 6 L), with valve and passivated interior surfaces.<sup>7</sup>

7.1.1.3 Vacuum/Pressure Gage,<sup>8</sup> Capable of measuring vacuum (–100 to 0 kPa or 0 to 30 in Hg) and pressure (0 to 200 kPa or 0 to 30 psig) in the sampling system. Gages shall be tested clean and leak tight.

7.1.1.4 Mass Flow Meter and Controller,<sup>9</sup> Capable of maintaining a constant flow rate ( $\pm 10\%$ ) over a sampling period of up to 24 h and under conditions of changing temperature (20 to 40°C) and humidity.

7.1.1.5 Filter, 7- $\mu\text{m}$  sintered stainless-steel in-line filter.<sup>10</sup>

7.1.1.6 Electronic Timer,<sup>11</sup> For unattended sample collection.

7.1.1.7 Solenoid Valve, Electrically operated, bi-stable solenoid valve with fluoroelastimer<sup>12</sup> seat and o-rings, or low temperature solenoid valve.

7.1.1.8 Tubing and Fittings, Chromatographic grade stainless steel tubing and fittings for interconnections. All such

materials in contact with sample, analyte, and support gases prior to analysis shall be chromatographic grade stainless steel.

7.1.1.9 Heater, thermostatically controlled to maintain temperature inside insulate sampler enclosure above ambient temperature if needed.<sup>13</sup>

7.1.1.10 Fan, For cooling sampling system, if needed.<sup>14</sup>

7.1.1.11 Thermostat<sup>15</sup>—Automatically regulates fan operation, if needed.

7.1.1.12 Maximum-Minimum Thermometer, Records highest and lowest temperatures during sampling period.<sup>16</sup>

7.1.1.13 Shut-Off Valve<sup>17</sup>—Stainless steel—leak free, for vacuum/pressure gage.

7.1.1.14 Auxiliary Vacuum Pump—optional, continuously draws 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. 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. Pump is not necessary if the intake manifold represents less than 5 % of the final sample.

7.1.1.15 Elapsed Time Meter<sup>18</sup>—To measure duration of sampling.

7.1.1.16 Optional Fixed Orifice, Capillary, Adjustable Micrometering Valve, or Vacuum Regulator, May be used in lieu of the electronic flow controller for grab samples or short duration time-integrated samples. Such systems require manual

<sup>7</sup> Scientific Instrumentation Specialists, Inc., P.O. Box 8941, Moscow, ID 83843, or Andersen Samplers, Inc., 4215-C Wendell Dr., Atlanta, GA 30336.

<sup>8</sup> Matheson, P.O. Box 136, Morrow, GA.

<sup>9</sup> Tylan Corp., 19220 S. Normandie Ave., Torrance, CA 90502.

<sup>10</sup> Nupro Co., 4800 E. 345th St., Willoughby, OH 44094.

<sup>11</sup> Paragon Elect. Co., 606 Parkway Blvd., P.O. Box 28, Twin Rivers, WI 54201.

<sup>12</sup> VITON® fluoroelastimer, trademark of E. I. du Pont de Nemours, has been found satisfactory. An equivalent can be used.

<sup>13</sup> Watlow Co., Pfafftown, NC.

<sup>14</sup> EG&G Rotron, Woodstock, NY, Model SUZAI.

<sup>15</sup> Elmwood Sensors, Inc., Pawtucket, RI.

<sup>16</sup> Thomas Scientific, Brooklyn Thermometer Co., Inc.

<sup>17</sup> Nupro Co., 4800 E. 345th St., Willoughby, OH 44094.

<sup>18</sup> Conrac, Cramer Div., Old Saybrook, CT.

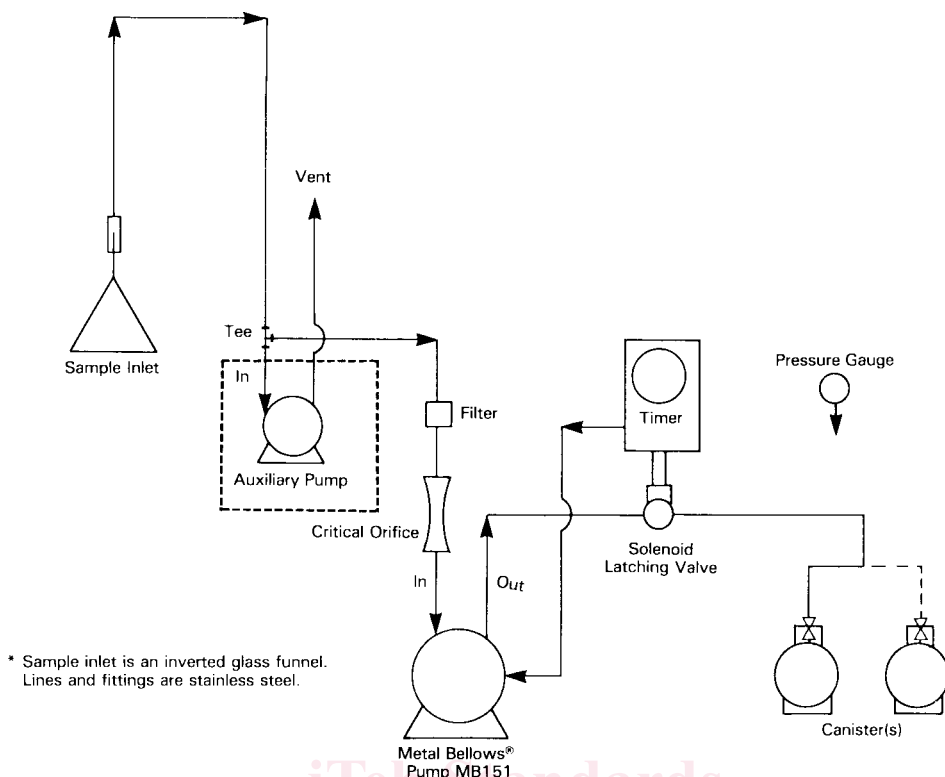


FIG. 2 Alternative Sampler Configuration for Pressurized Canister Sampling

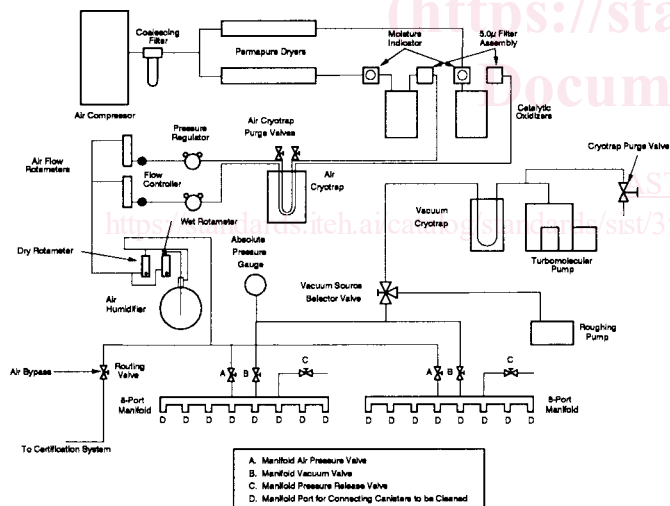


FIG. 3 Canister Cleanup Apparatus

activation and deactivation. In this standard, application of a pumpless simple orifice sampler is appropriate only in situations where samples consume 60 % of the total capacity of the canister used for collection. Typically this limits the sample duration to a maximum of 8 h per 6 L canister or 20 h per 15 L canister.

7.1.2 Pressurized—See Fig. 1 and Fig. 2.

7.1.2.1 Sample Pump, Stainless steel pump head, metal bellows type<sup>19</sup> capable of 200 kPa output pressure. Pump must

be free of leaks, clean, and uncontaminated by oil or organic compounds.

NOTE 2—Alternative sampling systems have been developed. The Rasmussen sampler (21) is illustrated in Fig. 2. 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. The Radian sampler (20) is illustrated in Fig. 3. This system draws air directly through a mass flow controller into an evacuated canister. Interferences using either configuration have been minimal.

7.1.2.2 Other Supporting Materials—All other components of the pressurized sampling system (Fig. 1, Fig. 2, and Fig. 3) are similar to components discussed in 7.1.1.1-7.1.1.16.

7.2 Sample Analysis Equipment:

7.2.1 GC/MS-Analytical System (Full Scan and SIM).

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

7.2.1.2 Gas Chromatograph, Capable of sub-ambient temperature programming for the oven, with other standard features such as gas flow regulators, automatic control of valves and integrator, etc. Flame ionization detector optional.

7.2.1.3 Chromatographic Detector, Mass spectrometric detector equipped with computer and appropriate software. 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 10.1.1.3 for complete description of trap and temperature control assembly. Traps may be built into the

<sup>19</sup> Metal Bellows Corp., 1075 Providence Highway, Sharon, MA 02067.