



Sorbent & Solution Sampling Guide

Table Of Contents

1.0 Introduction

2.0 Sorbent Sampling

2.1 Considerations for Sorbent Sampling	5
2.2 Method Specific Sampling Instructions	
EPA Method TO-4A	8
EPA Method TO-10A	10
EPA Method TO-13A High Volume	12
EPA Method TO-13A Low Volume	14
EPA Method 0010/8270C by MM5 Train	16
EPA Method TO-17	18
EPA Method VOST, 0030/5041A	22
NIOSH Methods	24

3.0 Solution Sampling

3.1 Method Specific Sampling Instructions	
EPA Method TO-5	26
EPA Method TO-11A	28
CARB 430 Method	30
EPA Methods 0011/316	32
Air Toxics Ltd. Method - Siloxanes	34

4.0 Filter Sampling

4.1 Method Specific Sampling Instructions	
EPA Method PM10 & TSP	36

1.0 Introduction

Air Toxics Ltd. presents this guide as a resource for air sampling. Air sampling can be more involved than water or soil sampling due to the reactivity of chemical compounds in the gas matrix and sample interaction with the sampling equipment and media. Ensuring that air samples are collected properly is an important step in acquiring meaningful analytical results. This guide is not a substitute for experience and cannot address the multitude of actual field conditions. Note that this guide is intended for typical projects involving sampling of volatile and semi-volatile organic compounds (VOCs and SVOCs) with sorbent tubes and impingers, and airborne particulates with filters. Air Toxics Ltd. also provides a “Guide to Air Sampling and Analysis – Canisters and Tedlar Bags” for whole air sampling of VOCs.

2.0 Introduction To Sorbent Sampling

Using a sorbent to collect an air sample normally involves “active” sampling, unlike an evacuated canister that can be filled “passively” by simply opening the valve. The most common method to draw an air sample through a sorbent device is to use a small pump with low flow rates between 10 to 200 milliliters per minute (mL/min) and tubing for connecting the components. For high volume applications, a high volume air sampler can be used with a larger sorbent cartridge.

Determining the Safe Sampling Volume

Determining the appropriate volume of air sample to draw through a sorbent tube is critical to achieving the data quality objectives. The tendency is to increase the sampling volume in order to lower the final reporting limit.



Note that over-sampling may saturate the sorbent tube and allow the target compound to breakthrough. *See method for volume guidelines.*



2.0 Introduction To Sorbent Sampling

It is imperative to know the flow rate through the sorbent tube and the sampling interval. A “set up” sorbent tube is often used to set the pump flow rate. It must be very similar to the actual sorbent tube being used for sample collection. The pressure drop through the sorbent tube determines the flow rate for a given pump setting. The flow rate through the sorbent tube should be monitored continuously (or at least periodically) using a rotometer or electronic flow sensor. If the sampling flow rate is greater than 200 mL/min, simple rotometers and electronic flow sensors cannot be used. It is necessary to use a device capable of measuring high flow rates. Refer to the method for information on the proper method of measuring the flow.

Common Sorbent Sampling Trains

The sampling train generally includes a sorbent tube, pump, and optional components such as a needle valve, particulate filter, and rotometer or electronic flow sensor. The sorbent tube is generally the first component in a sampling train, with the tubing and pump located downstream to minimize contamination of the sample.



The sorbent tube should be upstream of the pump.

For thermal desorption methods, it is critical that the air be drawn through the inlet side of the tube. The inlet side may be marked with a ring or the sampling direction may be indicated with an arrow. Proper orientation is especially important when using the multi-bed sorbent tubes described in EPA Method TO-17. If the low vapor pressure compounds are adsorbed on the high surface area sorbent (i.e., the one designed for gaseous compounds like vinyl chloride), they cannot be removed at the desorption temperatures routinely used.



For thermal desorption methods, the sorbent tube must be sampled in the appropriate direction.



2.0 Sorbent Sampling

Possible Components of a Sorbent Sampling Train

Tubing and Fittings: The components in the sampling train are connected with tubing and fittings as needed. Because tubing used to connect the sorbent tube to the pump does not come in contact with the air sample, the tubing material can be selected on its ability to seal. Tygon is an excellent material for this purpose, although Nylon and Teflon can be used. The length of tubing connecting the pump to the sorbent tube is not critical. Many sorbent tubes accept 1/4 in. Swagelok fittings and 1/4 in O.D. tubing.



Note that if the tubing is in the sample stream then it is necessary to select the appropriate grade material.

Needle Valve: If a variable speed/adjustable rate air sampling pump is not available, a needle valve can be used to adjust the flow rate. A rotometer (see the following section) with a built-in needle valve can provide an economical solution to adjust and measure sampling flow rate.

Rotometer or Electronic Flow Sensor: Although the flow rate for an air sampling pump can be calibrated before use, it is often desirable to include a flow measuring device in the sampling train. Note that the flow rate produced by a pump can decrease as more components are added to the sampling train. A rotometer is a relatively inexpensive meter that indicates flow with a small weight in a tapered column. As flow increases, air resistance of the weight increases and raises the weight until it is equilibrium with gravity. For best results, the rotometer must be kept vertical and free of particulates or moisture. An electronic flow sensor can provide more precise flow measurement than a rotometer, but is considerably more expensive. Both devices have optimum working ranges of flow rate (e.g., a typical rotometer may provide measurement from 100 to 500 mL/min).



Particulate Filter. If the air sample is known to have high levels of particulate matter, it may be necessary to place a filter at the sorbent tube inlet. In this case, the connecting tubing between the filter and the sorbent tube will be in the sampling stream and it should be new Teflon and as short as possible.

2.1 Considerations For Sorbent Sampling

Sorbent sampling requires the determination of the optimal sampling parameters: sample volume, flow rate and duration.

Appropriate sample volume should be determined by the media capacity and the required RLs. Matrix constituents such as water vapor and other non-target compounds should also be considered. See method specific sections for media capacity information and use the calculation provided to determine sample volume needed to meet specific RLs.

Determine the Final Reporting Limit for the Target Compound: the concentration may be a risk-based action level or EPA preliminary remediation goal (PRG).

Determine the Method Reporting Limit: the mass value is provided by the laboratory and is based on the analytical method selected and the sensitivity of the instrumentation. The method reporting limit may vary for each target compound.

Calculate the Sampling Volume: use the equation on the next page to determine the volume of air sample that must be drawn through the sorbent in order to achieve the final reporting limit.

Compare the Sampling Volume to the SSV: Refer to tables of safe sampling volume (SSV) for the sorbent being used – (e.g., Table 1 and Appendix 1 in Method TO-17). If the SSV for the compound of interest is not available, use the SSV of a compound in the same class (e.g., toluene for xylene, chloroform for carbon tetrachloride, etc.) and ensure that the compound will not breakthrough when sampling the volume calculated using the equation above. If breakthrough is a possibility, select a sorbent with greater sorbent strength (i.e., surface area).



2.1 Considerations For Sorbent Sampling

1) Calculate Minimum Sample Volume

$$\text{Minimum Volume (L)} = \frac{\text{Reporting Limit (ug)}}{\text{Action Level (ug/m}^3\text{)}} * \frac{1000 \text{ L}}{\text{m}^3}$$

Example: Screening Level = 0.08 ug/m³

$$\text{Minimum Volume (L)} = \frac{0.05 \text{ ug}}{0.08 \text{ ug/m}^3} * \frac{1000 \text{ L}}{\text{m}^3} = 625 \text{ Liters}$$

2) Calculate Minimum Flow Rate if time duration is set.

$$\text{Minimum Flow Rate (L/min)} = \frac{\text{Minimum Volume (L)}}{\text{Duration (min)}}$$

Example: TWA of 24 hours

$$\text{Minimum Flow Rate (L/min)} = \frac{625 \text{ L}}{24 \text{ hour} * 60 \text{ min}} = 0.44 \text{ L/min}$$

3) Calculate if Overloading of tube is possible.

For a source-impacted environment, estimate total concentration to estimate maximum volume. A safe sampling volume is considered to be 75% or less of the cartridge capacity.

Example: the standard TO-11A cartridge supplied by Air Toxics has a capacity of approximately 75 ug total carbonyls.

$$\text{Estimated maximum volume (L)} = \frac{(0.75 * 75 \text{ ug})}{\text{Est. Form. Conc (ug/m}^3\text{)}} * \frac{1000 \text{ L}}{\text{m}^3}$$

Example: Source-impacted site 3 ppmv (3700 ug/m³) Formaldehyde

$$\text{Estimated maximum volume (L)} = \frac{0.75 * 75 \text{ ug}}{3700 \text{ ug/m}^3} * \frac{1000 \text{ L}}{\text{m}^3} = 15 \text{ L}$$



2.1 Considerations For Sorbent Sampling

Avoid Over Sampling: The adage, “more is better”, often finds its way into sorbent sampling practice. In attempting to ensure low reporting limits, you may over sample a sorbent tube – especially if concentrations of target compounds are higher than expected.

Use a Backup Sorbent Tube: The use of a second, or “backup”, sorbent tube in series can help prevent compound breakthrough. Even if a safe sampling volume was calculated and not exceeded during sampling, a backup sorbent tube can provide insurance. The backup sorbent tube is only analyzed if a predetermined level of a given compound or total mass is found on the first tube. While the use of a backup sorbent tube will increase media costs, it usually has little effect on sampling costs. It will, however, provide definitive support for data integrity.



2.2 Method Specific Sampling Instructions

EPA Method TO-4A

Determination of Pesticides and Polychlorinated Biphenyls (PCBs) in Ambient Air using High Volume Polyurethane Foam (PUF) Sampling followed by Gas Chromatographic / Electron Capture Detector (GC/ECD)

<http://www.epa.gov/ttn/amtic/files/ambient/airtox/to-4a2r.pdf>

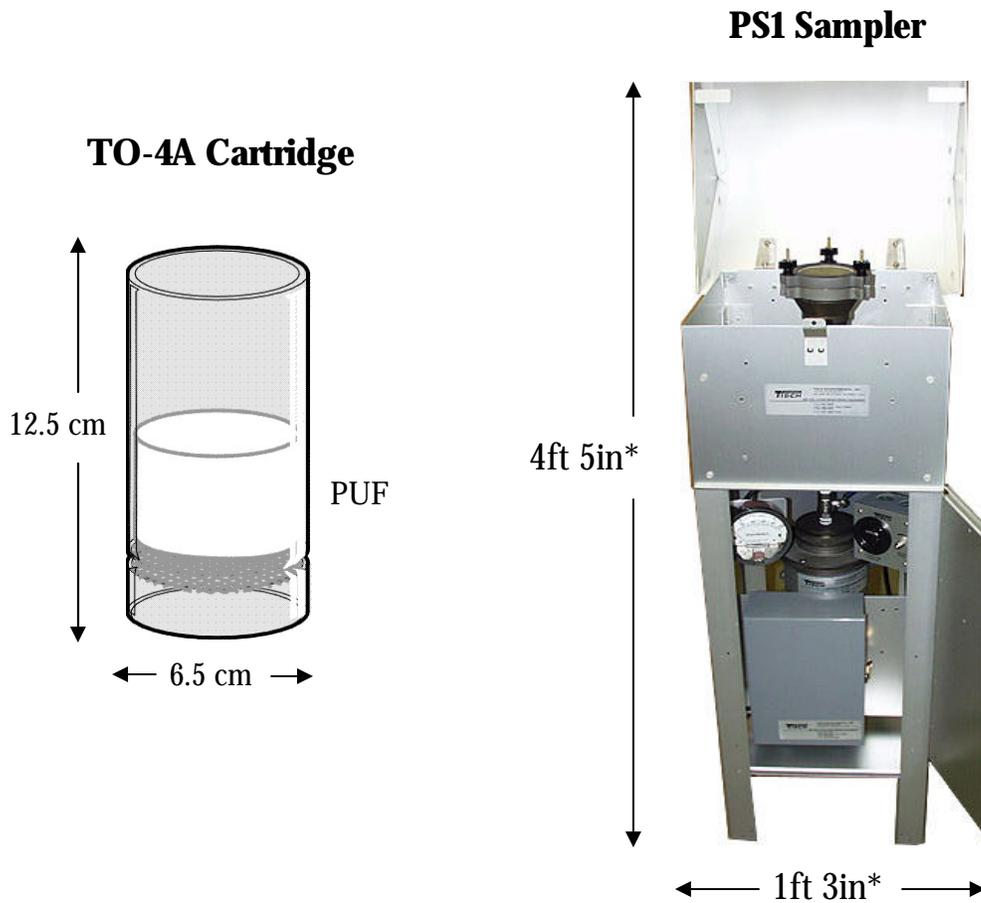
This method is applicable for the sample collection and analysis of pesticides and PCBs in ambient air. See pages 4A-32 and 4A-34 of the method for sampling diagrams.

TO-4A

Media	PUF Cartridge (high volume) with Filter (quartz fiber)
Type of Pump	High volume sampler; flow rate capability of (~10cfm)
Sampling Rate and Interval	0.225 m ³ /min for up to 24 hours
Sampling Volume	Up to 300 m ³ . Determined by user (as needed to achieve project reporting limits)
Sample Handling	Handle with aluminum foil and keep chilled at 4°C
Media Hold Time	30 days from date of media certification
Extraction Hold Time	7 days from sampling to extraction at 4°C
Sample Hold Time	40 days from extraction to analysis at 4°C
Field QC	At least 1 field blank; it is to be treated exactly like a sample except no air is drawn through the cartridge



2.2 Method Specific Sampling Instructions



Note: This method was developed using the PS-1 semi-volatile sampler provided by General Metal Works, Village of Cleves, OH as a guideline. EPA has experience in use of this equipment during various field monitoring programs over the last several years. Other manufacturers' equipment should work as well. However, modifications to these procedures may be necessary if another commercially available sampler is selected.

* Manufacturers include: Tisch Environmental, Anderson Instruments, and Thermo Environmental Instruments. Equipment specifications may vary by manufacture.



2.2 Method Specific Sampling Instructions

EPA Method TO-10A

Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air using Low Volume Polyurethane Foam (PUF) Sampling followed by Gas Chromatographic / Electron Capture Detector (GC/ECD)

<http://www.epa.gov/ttn/amtic/files/ambient/airtox/to-10ar.pdf>

This method is applicable for the sample collection and analysis of pesticides and PCBs in ambient air. See pages 10A-26 through 10A-28 of the method for sampling diagrams.

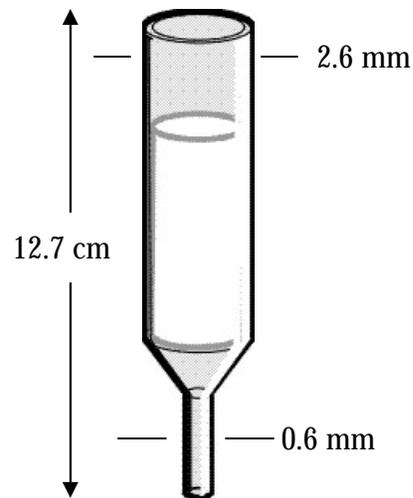
TO-10A

Media	PUF Cartridge (low volume) with or without filter
Type of Pump	Personal/portable continuous flow sampling pump; flow rate capability of 1-5 L/minute
Sampling Rate and Interval	1 to 5 L/min for 4 to 24hrs
Sampling Volume	Determined by user (as needed to achieve project RLs)
Sample Handling	Handle with aluminum foil and keep chilled at 4°C
Media Hold Time	30 days from date of media certification
Extraction Hold Time	7 days from sampling to extraction at 4°C
Sample Hold Time	40 days from extraction to analysis at 4°C
Field QC	At least 1 field blank; it is to be treated exactly like a sample except no air is drawn through the cartridge



2.2 Method Specific Sampling Instructions

TO-10A PUF Cartridge - Low Volume



2.2 Method Specific Sampling Instructions

EPA Method TO-13A

Determination of Polycyclic Aromatic Hydrocarbons (PAHs) in Ambient Air using Gas Chromatography / Mass Spectrometry (GC/MS)

<http://www.epa.gov/ttn/amtic/files/ambient/airtox/to-13arr.pdf>

This method is applicable for the analysis of PAHs in ambient air. The high volume PUF/XAD sampling procedure is applicable for a 24-hr sampling period. See pages 13A-55 and 13A-56 of the method for sampling diagrams.

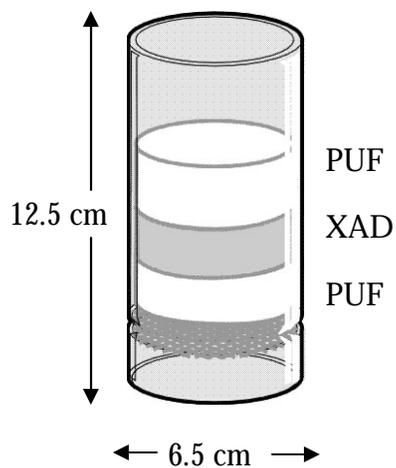
TO-13A

Media	PUF/XAD Cartridge (high volume) with Filter (quartz fiber)
Type of Pump	High volume sampler*; flow rate capability of (~10cfm)
Sampling Rate and Interval	0.225 m ³ /min for up to 24 hours
Sampling Volume	Up to 300 m ³ . Determined by user (as needed to achieve project reporting limits)
Sample Handling	Handle with aluminum foil and keep chilled at 4°C
Media Hold Time	30 days from date of media certification
Extraction Hold Time	7 days from sampling to extraction at 4°C
Sample Hold Time	40 days from extraction to analysis at 4°C
Field QC	At least 1 field blank; it is to be treated exactly like a sample except no air is drawn through the cartridge

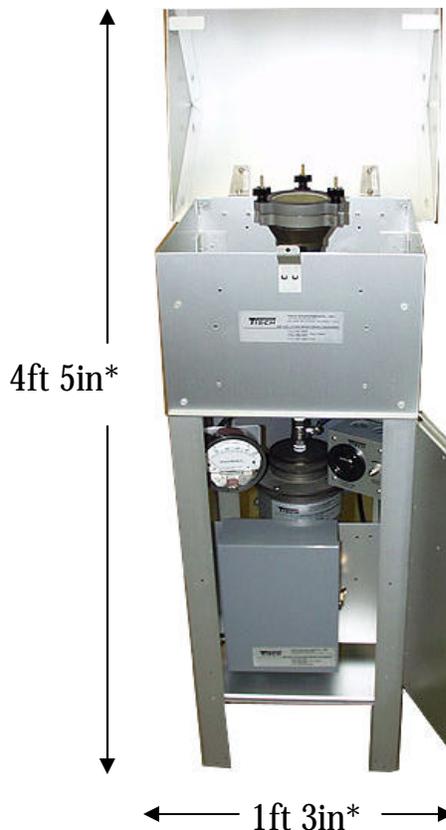


2.2 Method Specific Sampling Instructions

TO-13A PUF/XAD Cartridge



PS1 Sampler



Note: This method was developed using the PS-1 semi-volatile sampler provided by General Metal Works, Village of Cleves, OH as a guideline. EPA has experience in use of this equipment during various field monitoring programs over the last several years. Other manufacturers' equipment should work as well. However, modifications to these procedures may be necessary if another commercially available sampler is selected.

* Manufacturers include: Tisch Environmental, Anderson Instruments, and Thermo Environmental Instruments. Equipment specifications may vary by manufacture.



2.2 Method Specific Sampling Instructions

Modified EPA Method TO-13A Low Volume

Method TO-13A was intended for sampling SVOCs in ambient air and is generally unsuitable for indoor air applications. Placement of a high volume sampler in a home or office would be considered intrusive and impractical, at best. TO-13A can be modified such that the PUF and XAD-2 sorbent can be packed into a TO-10A cartridge or a XAD tube instead of the usual TO-13A cartridge. The TO-10A cartridge is packed with a combination of XAD-2 and PUF and may be used with or without a particulate filter (see Image 1). The XAD tube is packed with XAD-2 and also may be used with or without a filter (see Image 2). Both of these configurations are illustrated and can be provided by Air Toxics Ltd. on a project-specific basis, upon request.

Because of the pressure drop associated with having a packed bed of XAD-2 resin in the sampling tube, personal sampling pumps (e.g. normally used with small NIOSH sorbent tubes) may prove inadequate, given the large volume of air that must be sampled. These small pumps will have difficulty pulling flows greater than a few mL/min through the tube. A high capacity Kneuburger pump is powered by 12v – using a 12 volt converter or a 12 volt car battery (see Image 3).

Application	Media Type	Considerations
Indoor Air	PUF/XAD Cartridge - Low Volume	Desired volume to meet required reporting limits and desired duration of sampling i.e. 8hrs for an office or 12hrs for a home need to be calculated given the capacity of the car-
Soil Gas	XAD tube	Desired volume to meet required reporting limits considering the amount of vapor that can be collected from soil gas as well as the required flow rate of sampling need to be calculated given the capacity of the tube.



2.2 Method Specific Sampling Instructions

Image 1

PUF/XAD Cartridge - Low Volume

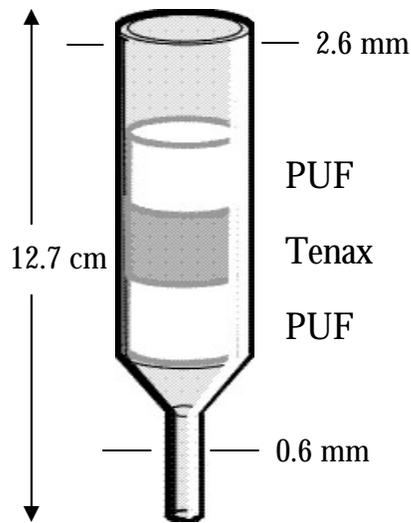


Image 2

XAD Tube

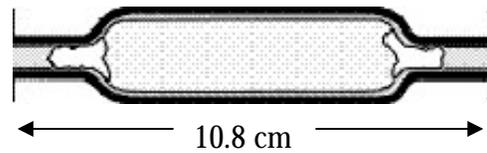
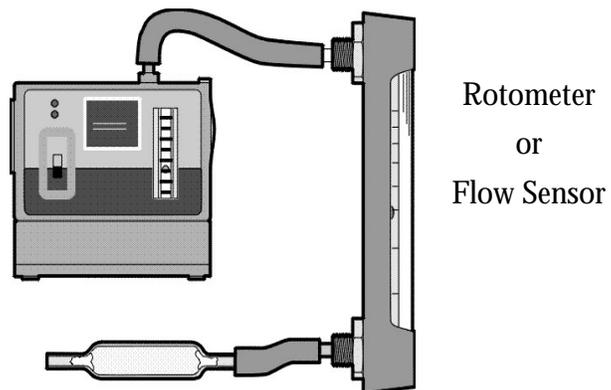


Image 3



2.2 Method Specific Sampling Instructions

EPA Method 0010/8270C

Modified Method 5 Sampling Train

Determination of Destruction and Removal Efficiency (DRE) of Semivolatile Principal Hazardous Compounds (POHCs) from Incineration Systems and Stationary Sources.

Method 0010 - <http://www.epa.gov/epaoswer/hazwaste/test/under.htm>

Method 8270C - http://www.epa.gov/epaoswer/hazwaste/test/8_series.htm

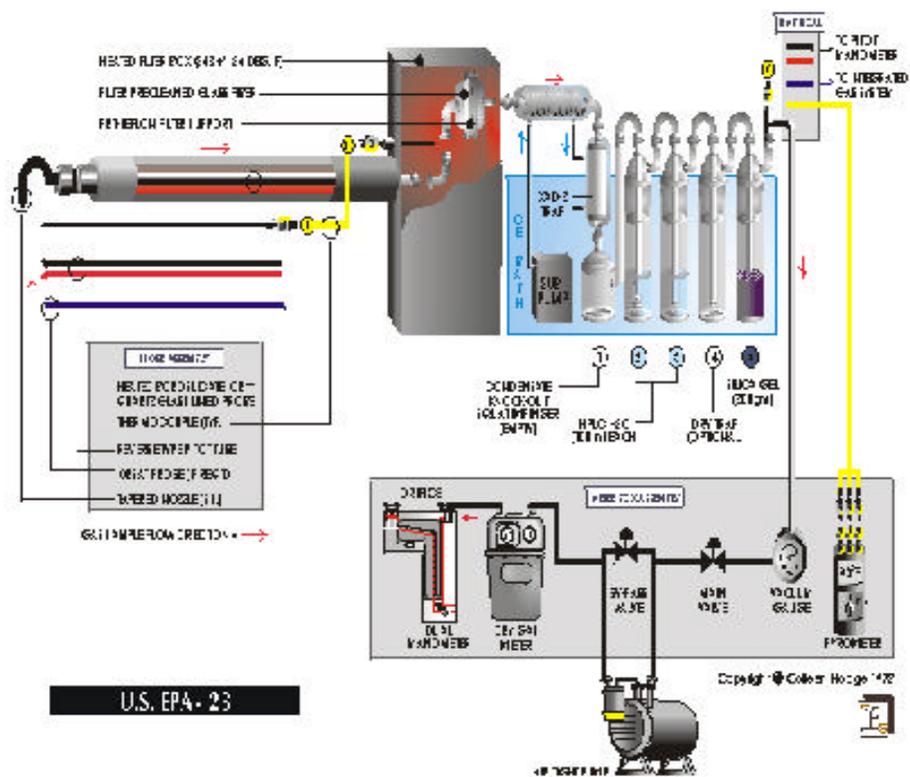
0010/8270C Modified Method 5 Sampling Train

Media	Filter (quartz fiber) and sorbent trap (20 g XAD-2)
Type of Pump	Metering system. Pump capable of 4 cfm free flow. see section 4.1.3.9 of method 0010
Sampling Rate and Interval	Determined by user (isokinetic)
Sampling Volume	Determined by user (3 dscm min or as needed to achieve project reporting limits)
Sample Handling	Wrap XAD-2 trap in aluminum foil, and keep chilled at 4°C
Media Hold Time	30 days from date of media certification
Extraction Hold Time	14 days from sampling to extraction at 4°C
Sample Hold Time	40 days from extraction to analysis at 4°C
Components of the Sample Train	1. Probe 2. Heated Filter 3. XAD-2 Sorbent Trap 4. Impingers 5. Condensates



2.2 Method Specific Sampling Instructions

A typical MM5 sampling train



2.2 Method Specific Sampling Instructions

EPA Method TO-17

Determination of Volatile Organic Compounds in Ambient Air using Active Sampling Onto Sorbent Tubes.

<http://www.epa.gov/ttn/amtic/files/ambient/airtox/to-17r.pdf>

This method replaces earlier sorbent-based EPA Methods TO-1 and TO-2 and provides an alternative to canister-based EPA Method TO-15. The target compound list is the same as TO-15 (i.e., subsets of the 97 VOCs listed as hazardous air pollutants in the Clean Air Act Amendments of 1990). However, TO-17 can collect VOCs over a wider volatility range than TO-15, by using a tube with multiple sorbents packed in increasing sorbent strength. Both single and multi-bed sorbent tubes are described in TO-17. Tube Style 3 (i.e., Carbotrap 300 by Supelco) can be used for compounds ranging in volatility from n-C₃ to n-C₁₆ for air volumes of 2 L at relative humidity below 65% and temperatures below 30°C. Volumes greater than 5 L can be collected, but C₃ compounds are not quantitatively retained. Single bed tubes, such as Tenax TA, can be used to effectively collect Naphthalene and middle distillate fuels in indoor air.



Media	Sorbent tube (e.g., Carbotrap 300) with optional particulate filter and ozone scrubber
Type of Pump	Low flow rate pump (10 to 200 mL/min) or high flow pump with low flow adapter
Sampling Rate and Interval	17 and 67 mL/min for 1 hour (example flow rates)
Sampling Volume	1 and 4 L (example volumes not mandated)
Sample Handling	Cap ends, place in culture tube, keep chilled at 4°C
Media Hold Time	Not specified - recommend 30 days at 4°C
Sample Hold Time	30 days from collection at 4°C
Field QC	Field Blanks - two per sampling event
Distributed Pair	One location sampled at two volumes



2.2 Method Specific Sampling Instructions

Although the method suggests sampling volumes of 1 and 4 L, there is considerable mention of calculating a safe sampling volume. The sampling volume you select should include consideration of both the desired final reporting limit and the safe sampling volume of the sorbent being used (see Section 2.1). The thought process is outlined below.

Determine the Final Reporting Limit for the Target Compound: the concentration may be a risk-based action level or EPA preliminary remediation goal (PRG).

Determine the Method Reporting Limit: the mass value is provided by the laboratory and is based on the analytical method selected and the sensitivity of the instrumentation. The method reporting limit may vary for each target compound.

Calculate the Sampling Volume: use the equation on page 6 of this guide to determine the volume of air sample that must be drawn through the sorbent in order to achieve the final reporting limit.

Compare the Sampling Volume to the SSV: Refer to tables of safe sampling volume (SSV) for the sorbent being used – (e.g., Table 1 and Appendix 1 in Method TO-17). If the SSV for the compound of interest is not available, use the SSV of a compound in the same class (e.g., toluene for xylene, chloroform for carbon tetrachloride, etc.) and ensure that the compound will not breakthrough when sampling the volume calculated using the equation above. If breakthrough is a possibility, select a sorbent with greater sorbent strength (i.e., surface area).

To illustrate the thought process, an example is provided below:

1. Assume Benzene is the target compound and must be reported at a final reporting limit of 0.0005 ug/L.
2. The laboratory provides a method reporting limit of 10 ng (0.010 ug).
3. Using Equation 3, the sampling volume = $0.010/0.0005 = 20$ L.
4. TO-17 Appendix 1 shows that for Benzene, a SSV of up to 26 L can be collected using a Type 3 (CarboTrap 300) multi-sorbent tube.

In this example, Benzene can theoretically be reported at a final reporting limit of 0.0005 ug/L with a sampling volume of 20 L.



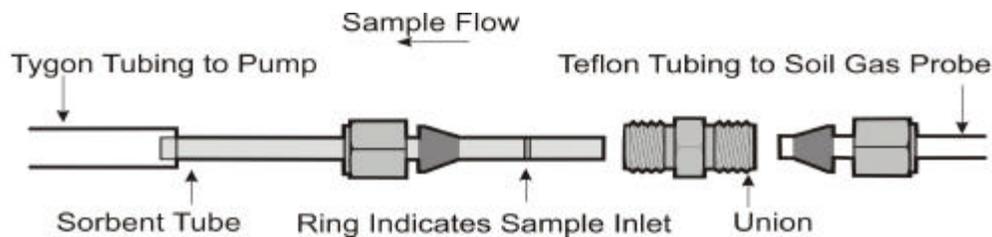
2.2 Method Specific Sampling Instructions

TO-17 Sampling Instructions

Application: Soil Gas

Media: TO-17 tube, 1/4" Teflon tubing, 1/4" Tygon tubing, 1/4" to 1/4" Union, 1/4" fittings with ferrules, a sample pump and a low flow holder maybe required if using a higher flow pump

Typical Sampling Parameters: **Sample Flow Rate** = 50mL/min **Total Vol.** = 200 mL **Duration** = 4 min. These parameters may change depending on project objectives.



Instructions:

- 1) In order to calibrate the pump use a "set-up" tube. Using the Tygon tubing connect the sampling pump to the outlet of the sorbent tube, if using a higher flow pump a low flow holder may be necessary to lower the flow rate, then connect the inlet (the ringed side) to the calibrator. Adjust setting to desired flow rate and record.
- 2) Replace the "set-up" tube with a sample tube. Again using the Tygon tubing connect the sampling pump to the outlet of the sample tube. Attach the inlet to the union fitting using a Swagelok nut. Using a 9/16" wrench on the nut and a 7/16" wrench on the union, tighten the nut. In the same manner, attach the union to the Swagelok nut on the soil gas probe tubing. DO NOT OVERTIGHTEN.
- 3) Start the sample pump and record the start time. After the desired duration, stop the pump and record the end time.
- 4) Replace the end plugs on both ends of the sample tube. Record the sample ID, tube ID and the collection date/time on the COC.
- 5) When completed with a set of samples, re-attach the "set-up" tube to the calibrator and measure the post-sampling flow. Record post-sampling flow rate. This should match within 10% of the pre-sample flow rate.
- 6) Record sample volume on the COC using the average of the pre- and post- flow rates.
- 7) Send tubes to the lab in the cooler with ice.



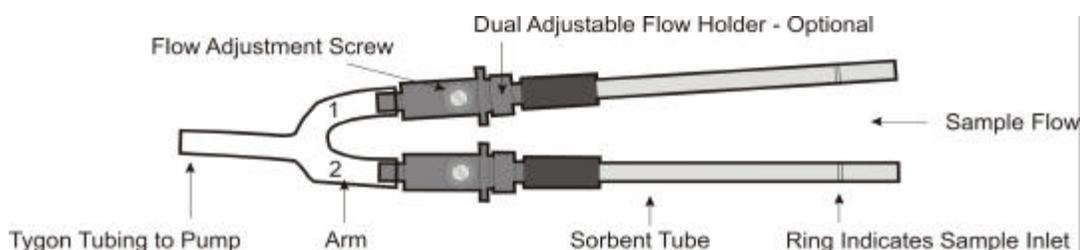
2.2 Method Specific Sampling Instructions

TO-17 Sampling Instructions

Application: Indoor Air

Media: TO-17 tube, 1/4" Tygon tubing, sample pump & optional dual adjustable flow holder

Parameters: Typical flows should be between 10 to 200 ml/min. Consult with the laboratory to insure appropriate volumes are collected to meet desired reporting limits.



Instructions:

- 1) Connect the sampling pump to the outlet of a "set-up" tube using Tygon tubing, connect the inlet (the ringed side) to the calibrator. Adjust setting to desired flow rate and record. A low flow holder may be required for a higher flow pump.
- 2) Replace the "set-up" tube with a sample tube. Using the Tygon tubing connect the sampling pump to the outlet of the sample tube.
- 3) The picture above shows a distributed pair using an adjustable 2-tube flow holder. This allows you to take replicate or distributed samples. The flow is adjusted by tightening the screw on the holder. Two different flows can be used to collect two volumes for a distributed pair. A 2-tube holder is not necessary for single sample collection. If using a dual holder it is important to notate which arm corresponds to each recorded flow measurement.
- 4) Start the sample pump and record the start time. After the desired duration, stop the pump and record the end time.
- 5) Replace the end plugs on both ends of the sample tube. Record the sample ID, tube ID and the collection date/time on the COC.
- 6) When completed with a set of samples, re-attach the "set-up" tube to the calibrator and measure the post-sampling flow. Record post-sampling flow rate. This should match within 10% of the pre-sample flow rate.
- 7) Record sample volume on the COC using the average of the pre- and post- flow rates.
- 8) Send tubes to the lab in the cooler with ice.



2.2 Method Specific Sampling Instructions

EPA Method 0030/5041A (VOST)

This method describes the collection of volatile principal organic hazardous constituents (POHCs) from the stack gas effluents of hazardous waste incinerators. For the purpose of definition, volatile POHCs are those POHCs with boiling points less than 100°C. If the boiling point of a POHC of interest is less than 30°C, the POHC may break through the sorbent under the conditions of the sample collection procedure.

ATL has validated a method of “diluting” a VOST sample prior to analysis in the case of high level matrices. The first step is to inject surrogate benzene-d6 onto the sample tube(s). Recovery is monitored as a means of ensuring the integrity of the sample transfers. The VOST tubes are then thermally desorbed into a Tedlar bag. This gaseous sample is screened using GC/FID and an acceptable amount of sample is transferred back onto a clean set of VOST tubes which are subsequently thermally desorbed per the method. High quality data is obtained from every tube.

Method 0031

M0031 was written in part to specify which organic compounds are appropriate for collection in contrast to M0030 which provided guidelines based on boiling point alone. Moreover, polar and reactive compounds are identified as poor performers for the M0031 collection strategy. In Method 0031 the VOST consists of three glass tubes containing Tenax, Tenax, and Anasorb, respectively, to trap the organic mass.

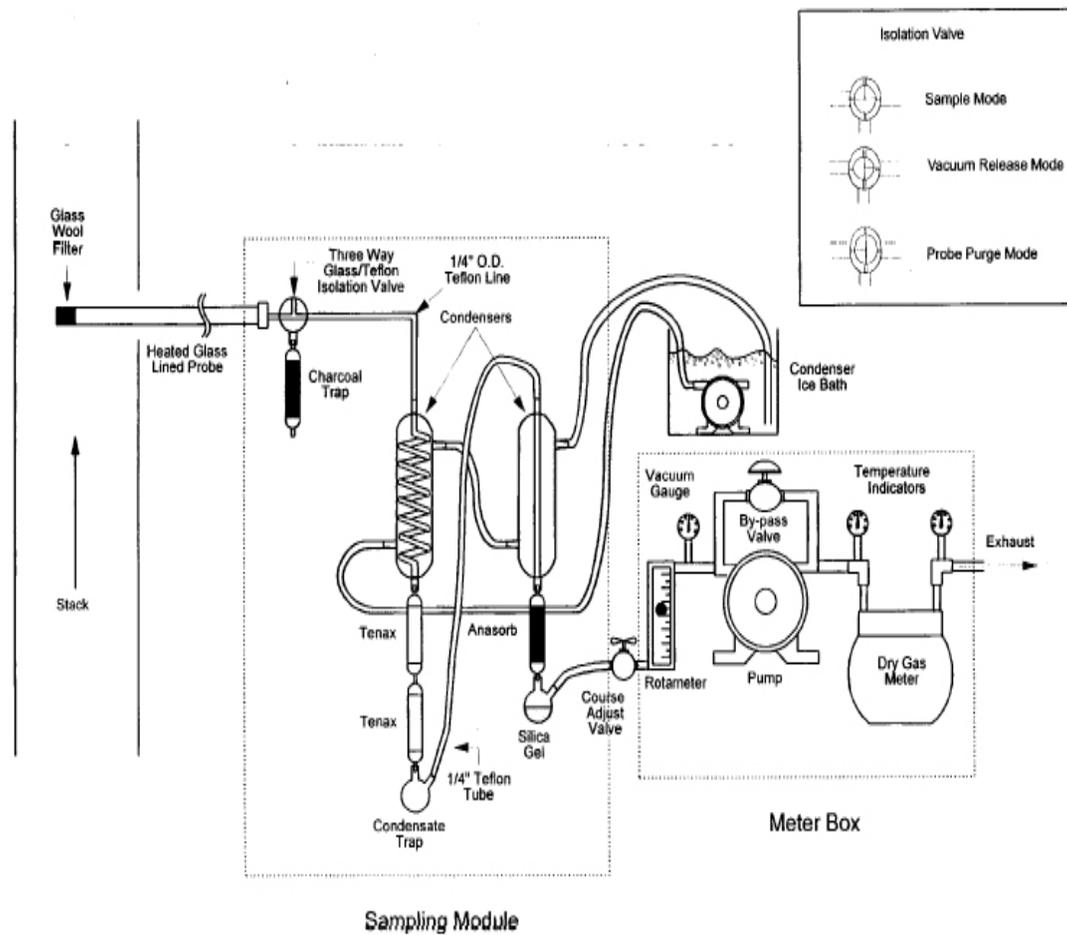
0030/5041A VOST

Media	One pair of glass sorbent tubes (1.6 g Tenax; 1.0 g Tenax / 1.0 g petroleum based charcoal)
Media Hold Time	2 weeks at 4°C
Type of Pump	see method for sampling equipment
Sampling Rate/Interval	1 L/min for 20 min (FAST-VOST); 250 mL/min for 20 min and 500 mL/min for 40 min (SLOW-VOST)
Sampling Volume	20 L (FAST-VOST); 5 and 20 L (SLOW-VOST)
Sample Handling	Cap ends, place in culture tube, keep chilled at 4°C
Hold Time to Analysis	14 days at 4°C



2.2 Method Specific Sampling Instructions

VOST Sample Train



2.2 Method Specific Sampling Instructions

NIOSH 1500/1501

Media	Coconut Shell Charcoal tube, 100mg/50mg tube
Media Hold Time	Use manufactures expiration date listed on tube
Type of Pump	Personal Sampling Pump
Sampling Rate	0.01 - 0.2 L/min
Sampling Volume	Ranges from a minimum of 1L to a maximum of 30L Dependant on contaminant; see method
Sample Handling	Ship cold after sampling
Sample Hold Time	30 days at 5°C
Field QC	10% field blanks

NIOSH 1550

Media	Coconut Shell Charcoal tube, 100mg/50mg tube
Media Hold Time	Use manufactures expiration date listed on tube
Type of Pump	Personal Sampling Pump
Sampling Rate	0.01 - 0.2 L/min
Sampling Volume	Minimum - 1.3 L; Maximum - 20 L
Sample Handling	Ship cold after sampling
Hold Time to Ex- traction	7 days at 25°C
Extract Hold Time	2 to 10 field blanks per set



2.2 Method Specific Sampling Instructions

NIOSH 5515

Media	5515 filter + Sorbent (37 mm PTFE + 100mg/50mg XAD-2 tube)
Media Hold Time	Use manufactures expiration date listed on tube
Type of Pump	Personal Sampling Pump, capable of operating up to 8hrs at 2 L/min
Sampling Rate	2 L/min
Sampling Volume	Minimum - 200 L; Maximum - 1000 L
Sample Handling	Handle with aluminum foil and keep chilled at 4°C and keep out of sunlight
Sample Hold Time	Unspecified; ATL uses 7 days
Field QC	2 to 10 field blanks; 6 to 10 media blanks

NIOSH 5503

Media	5503 filter + Sorbent (13 mm glass fiber + 100mg/50mg
Media Hold Time	Use manufactures expiration date listed on tube
Type of Pump	Personal Sampling Pump
Sampling Rate and Interval	0.05 - 0.2 L/min
Sampling Volume	Minimum - 1 L; Maximum - 50 L
Sample Handling	Handle with aluminum foil and keep chilled at 4°C and keep out of sunlight
Hold Time	Unspecified for filter; ATL uses 7 days. Method allows 2 months for Florisil tube
Field QC	2 to 10 field blanks per set



3.0 Solution Sampling

The gas can be drawn (bubbled) through a solution in an impinger to dissolve the compounds in the gas phase. The sampler records the flow rate and sampling interval for calculating compound concentration by volume. The impinger vial is subsequently capped, chilled (as required), and transported to the laboratory for analysis. Sampling with a solution is similar to sorbent sampling in that compounds are concentrated and the gas matrix is not collected. However, the solution is chosen so that the compounds of interest are either dissolved (and consequently remain in solution) or form a derivative in-situ (and the derivative remains in solution). The compounds/derivatives are analyzed directly or by chemical extraction. The solution (e.g., methanol, DNPH, water) is selected according to the type of air sampling and compounds targeted. An acidic solution of dinitrophenylhydrazine (DNPH) is used to derivatize C1-C3 carbonyl compounds.

3.1 Method Specific Sampling Instructions

EPA Method TO-5

Method for the determination of aldehydes and ketones in ambient air using High Performance Liquid Chromatography (HPLC). <http://www.epa.gov/ttn/amtic/airtox.html>

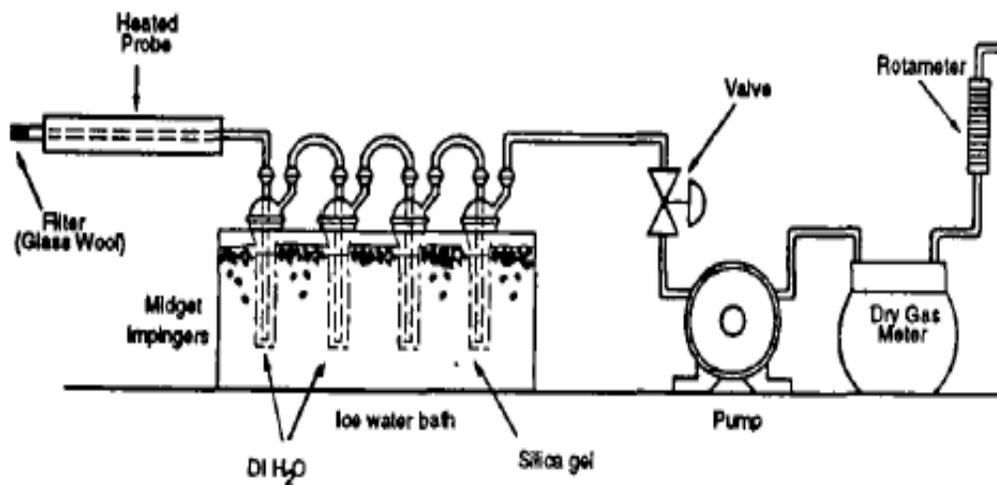
TO-5

Media	One pair of 40 mL glass vials with Teflon screw caps and 25 mL midget impingers in ice bath
Impinger Solution	10 mL 2,4-dinitrophenylhydrazine (DNPH) and 10 mL iso-octane
Sampling Volume	Up to 80 L
Sampling Rate	100 to 1,000 mL/min
Sample Handling	Cap vials and keep chilled at $4 \pm 2^{\circ}\text{C}$
Media Hold Time	48 hrs from date of media preparation at 4°C
Extraction Hold Time	7 days from sampling to extraction at 4°C
Sample Hold Time	30 days from extraction to analysis at 4°C



3.1 Method Specific Sampling Instructions

Sampling Train



- Front and Back impingers are to be combined at the laboratory.
- Iso-octane can be used

Field QC:

Field Blank—At least one FB with each group of samples. FB is treated identically to the samples except that no air is drawn through the reagent.



3.1 Method Specific Sampling Instructions

EPA Method TO-11A

Formaldehyde and other carbonyl compounds (aldehydes and ketones) in air are collected by drawing sample through a DNPH-coated silica gel cartridge using a sampling pump. TO-11A can be applied to indoor air, ambient air, and source-impacted sites. Collection times up to 24 hours are used for low ppbv environments, and short-term sampling (5 to 60 minutes) can be used for higher concentration sites.

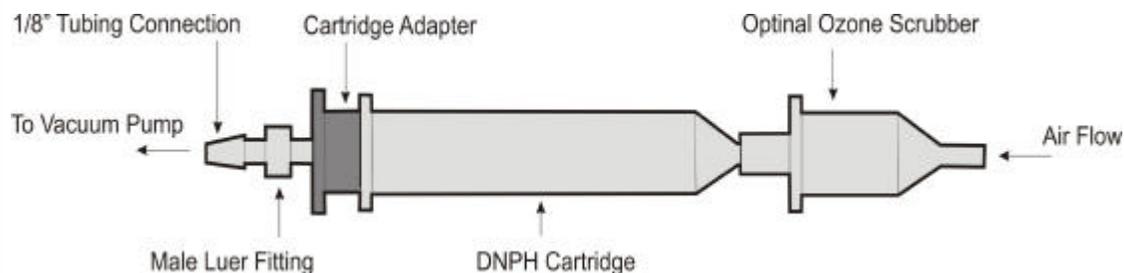
<http://www.epa.gov/ttn/amtic/airtox.html>

TO-11A

Media	Sep-Pak cartridges (DNPH-coated silica gel) an ozone scrubber is strongly recommended
Sampling Rate	Range: 0.1 to 2 L/min. Typical rate for ambient air <1 L/min when using a personal sampling pump.
Cartridge Capacity	S10 Supelco = Approximately 75 ug total carbonyls. Cartridges with higher capacity are available. If breakthrough is a concern, use a back-up tube.
Sample Handling	Cap ends, place in foil-lined envelope included in shipment. Label envelope with sample information. Keep chilled at ~ 4°C and keep out of sunlight.
Media Hold Time	Manufacturer's expiration date listed on cartridge
Sample Hold Time	14 days
Field QC Samples	Field Blank – Treat in the same manner as samples, but do not draw air through cartridge. Field Duplicate – Collect a collocated sample using a second sampling port attached to the sample pump. Back-up tube – If breakthrough is a concern, a back-up tube can be connected to the sample tube.
Sampling QC	QC Measure and record the flow rate before and after sample collection. Flow rates should not vary more than 10% over the sampling duration.



3.1 Method Specific Sampling Instructions



Interferences: Atmospheric ozone can result in a loss of formaldehyde and other carbonyl derivatives. An ozone scrubber is recommended to minimize interference. Particulate-laden atmospheres ($>50 \text{ ug/m}^3$) may result in flow drops during sampling. Additionally, acrolein and crotonaldehyde may partially degrade using DNP-coated silica gel cartridges.

Special Considerations: Compound breakthrough can occur if too much volume is collected and the sorbent becomes overloaded. If breakthrough is a concern, cartridges may be sampled as a train. The two cartridges are analyzed separately by the laboratory to monitor breakthrough.



3.1 Method Specific Sampling Instructions

CARB Method 430

This method applies to the determination of formaldehyde and acetaldehyde emissions from stationary sources. The method is based on the use of high performance liquid chromatography (HPLC).

<http://www.arb.ca.gov/testmeth/vol3/vol3.htm> See sections 8.0 - 8.2 on pages 18 - 23 for sampling procedures.

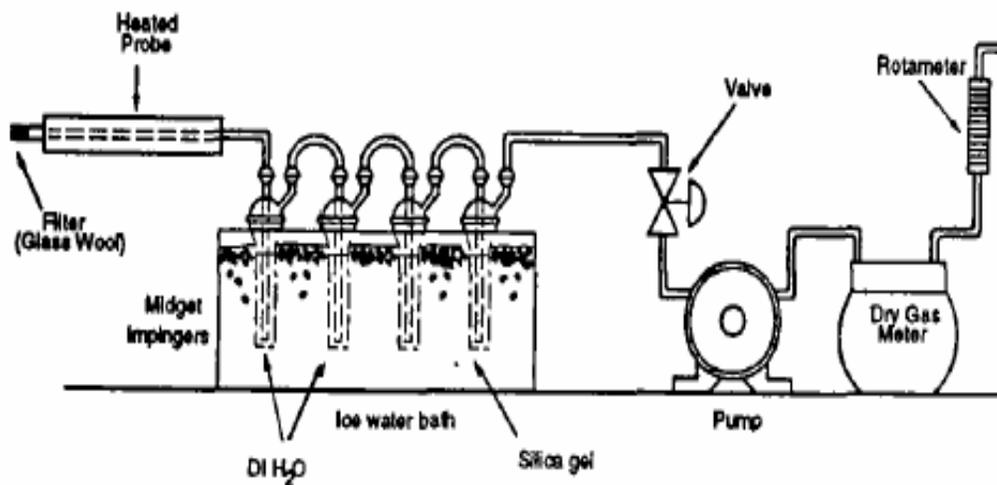
CARB 430

Media	One pair of 25 mL glass vials with Teflon screw caps and 35 mL midget impingers in ice bath
Impinger Solution	10 mL 2,4-dinitrophenylhydrazine (DNPH)
Sampling Volume	Up to 80 L
Sampling Rate	100 to 1,000 mL/min
Sample Handling	Cap vials and keep chilled at $4 \pm 2^{\circ}\text{C}$
Media Hold Time	48 hrs from date of media preparation at 4°C
Extraction Hold Time	7 days from sampling to extraction at 4°C
Sample Hold Time	30 days from extraction to analysis at 4°C



3.1 Method Specific Sampling Instructions

Sampling Train



- Each vial will be treated as an individual sample, do not combine.
- Iso-octane should not be used.

Field QC:

Trip Blank

Field Blank

Field Spike (as requested)



3.1 Method Specific Sampling Instructions

EPA Method 0011 Sampling for selected aldehyde and ketone emissions from stationary sources. <http://www.epa.gov/epaoswer/hazwaste/test/under.htm>

Method 0011 sampling is comprised of an isokinetic sampling train containing DNPH filled impingers. Formaldehyde and other aldehydes and ketones react with the DNPH to form derivatives. The derivatives are extracted and analyzed by HPLC/UV.

Method 0011

Media	DNPH filled impingers
Sampling Rate	Determined by user (isokinetic)
Sampling Volume	Determined by user
Sample Handling	Cap vials and keep chilled at $4 \pm 2^{\circ}\text{C}$
Media Hold Time	5 days from date made and 2 days from date opened in the field
Extraction Hold Time	7 days from sampling to extraction at 4°C
Sample Hold Time	30 days from extraction to analysis at 4°C
Quality Control	<p>Field Spike - typically ~ 800 ug of formaldehyde. (Place the field spike into an impinger containing 200 mL of DNPH solution, recover, and return for analysis.)</p> <p>Sample Blank - add a volume of DNPH reagent and Methylene chloride equal to the total volume of the combined impingers and rinses.</p> <p>Matrix Spike Sample - this additional sample may be collected for use as a matrix spike sample as described in Sec. 8.0 of Method 8315.</p> <p>Field blanks - may be submitted with the samples collected at each sampling site.</p>



3.1 Method Specific Sampling Instructions

EPA Method 316

Sampling and Analysis for Formaldehyde Emissions from Stationary Sources in the Mineral Wool/Wool Fiberglass Industries. <http://www.epa.gov/ttn/emc/promgate.html>

Method 316 sampling is comprised of an isokinetic sampling train containing reagent grade water filled impingers. Formaldehyde is absorbed in the DI water. The contents of the impinger and DI train rinses are analyzed for by a modified pararosaniline method. Samples are derivatized with pararosaniline and sodium sulfite to create a purple chromophore. The chromophore is analyzed by spectrophotometer set at 570nm.

EPA Method 316

Media	Reagent grade water filled impingers
Sampling Rate	Determined by user (isokinetic)
Sampling Volume	30 cubic ft minimum sample volume
Sample Handling	Cap vials and keep chilled at 2°C
Sample Hold Time	14 days from date of collection at 2°C
Field Blank	1 per set



3.1 Method Specific Sampling Instructions

Air Toxics Method @ 71 Siloxanes

Siloxanes are a family of organic compounds containing chains of silicon, oxygen, and methyl groups. These organosilicon compounds, commonly called silicones, differ from naturally occurring inorganic forms of silicon (i.e., silicates). Siloxanes are manufactured in a wide variety of forms including low to high viscosity fluids, gums, elastomers, and resins.

Building on results of the 1997 Dow Corning landfill consortium investigation, the ATL method is based on drawing air-phase samples through a series of two midjet impingers containing methanol (see Table 1). Siloxanes present in the air-phase dissolve in the chilled methanol solution and are subsequently capped and kept chilled until analysis. The suggested media hold time is 30 days and the suggested sample hold time until analysis is 21 days.

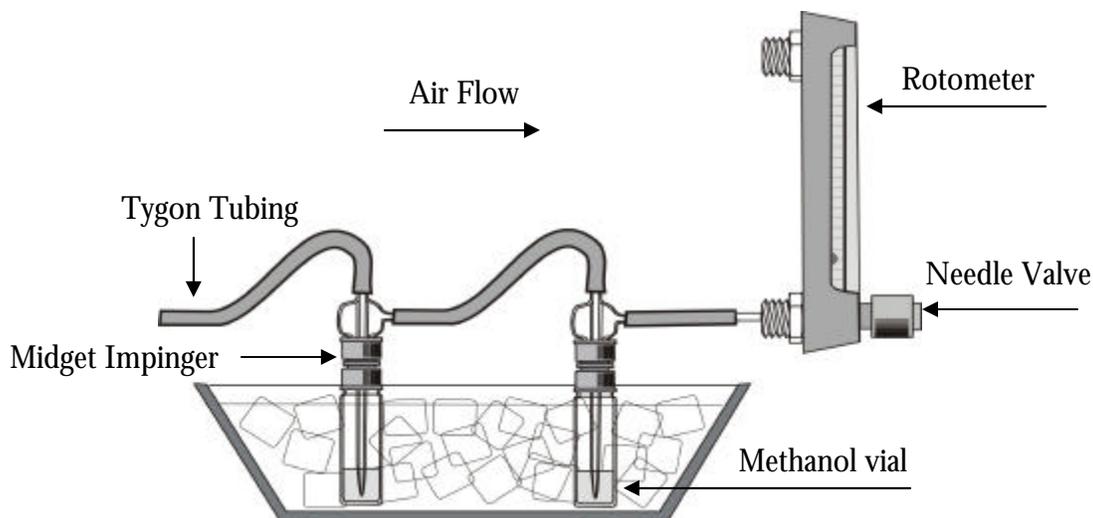
Air Toxics @ 71 Siloxanes

Media	One pair of 24 mL borosilicate glass vials with Teflon screw
Impinger Solution	Up to 15 mL methanol (6 mL suggested)
Sampling Volume	Determined by user (20 L suggested)
Sampling Rate	Determined by user (112 mL/min for 3 hours suggested)
Sample Handling	Cap vials and keep chilled at $4 \pm 2^{\circ}\text{C}$
Media Hold Time	30 days from date of certification
Sample Hold Time	21 days from collection



3.1 Method Specific Sampling Instructions

Collect the sample by attaching inert, flexible tubing from the source air stream to the inlet of the first impinger (see Figure 4). Additional tubing connects the outlet of the first impinger to the inlet of the second impinger and both impingers are chilled in an ice bath. If the source is not under pressure, a low-volume pump can supply the vacuum required to draw the sample through the impingers.



A needle valve and rotameter can be used to adjust and measure the flow rate of sample through the impingers. The user must determine optimum sampling rate and volume to achieve the data quality objectives of the sampling program. Sampling rates from 100 to 1,000 mL/min are appropriate as long as there is not significant loss of impinger solution. The amount of sample air drawn through the impingers and the amount of methanol in the impinger determine the final reporting limit concentration. The more sample air drawn through the impingers equates to more target constituent concentrated in the solution and thus lower reporting limits. Be careful not to over sample and saturate the solution. Less impinger solution equates to lower reporting limits, but has less capacity to dissolve the target constituents. For applications involving siloxanes removal from methane gas sources, Applied Filter Technology suggests filling each impinger with 6 mL of methanol and sampling at a flow rate of 112 mL/min for 180 minutes [4]. This arrangement results in a sampling volume of approximately 20 L.



4.0 Filter Sampling

Gas may be drawn through a filter to immobilize the compounds suspended in a gas matrix (or adsorbed on particulates suspended in a gas matrix) onto the surface of the filter material. The sampler records the flow rate and sampling interval for calculating compound concentration by volume. The filter is bagged, chilled (as required), and transported to the laboratory for analysis. Sampling with a filter is similar to sorbent and solution sampling in that compounds are concentrated and the gas matrix is not collected. The filter may be weighed before and after sampling to provide a gravimetric analysis of the total particulates and compounds collected. In addition, the filter can undergo solvent extraction or digestion to determine the presence of organic and inorganic compounds. The filter (e.g., quartz fiber, mixed cellulose ester, Teflon) is selected according to the type of air sampling and compounds targeted.

4.1 Method Specific Sampling Instructions

PM10

This method provides for the measurement of the mass concentration of particulate matter with an aerodynamic diameter less than or equal to a nominal 10 micrometers (PM10) in ambient air over a 24-hour period for purposes of determining attainment and maintenance of the primary and secondary national ambient air quality standards for particulate matter.

<http://www.epa.gov/ttn/emc/promgate.html>

TSP

See PM10

The methods for TSP and PM-10 differ slightly in the conditioning requirements for the filters. Air Toxics treats the two methods the same way using the more stringent conditioning requirements.



4.1 Method Specific Sampling Instructions

PM10 & TSP

Media	8 X 10 inch glass fiber filter
Media Hold Time	None specified.
Type of Pump	High volume sampler*
Sampling Rate and Interval	0.5 m ³ /min for 24 hours
Sampling Volume	700 m ³
Sample Handling	None Specified. Use protective packaging. Do not allow the filters to get wet or damaged. Do not handle with bare
Hold Time to Analysis	Air Toxics suggests 14-day sample hold time.
Analytical Method	Gravimetric
QC Samples	Duplicates, Lab Blank, Trip Blank



* Manufacturers include: Tisch Environmental, Village of Cleves-Ohio; Anderson Instruments-500 Technology Ct, Smyrna, GA; and Thermo Environmental Instruments-8 West Forge Parkway, Franklin, MA.

