# U.S. EPA BASE STUDY
# STANDARD OPERATING PROCEDURE
# FOR SAMPLING
# VOLATILE ORGANIC COMPOUNDS
# IN INDOOR AIR
# USING MULTISORBENT SAMPLERS

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Prepared By:

**Environmental Health & Engineering, Inc.**
**60 Wells Avenue**
**Newton, MA 02459-3210**

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# 1.0 OBJECTIVE

The objective of this procedure is to collect representative samples of volatile organic compound (VOC) contaminants present in indoor and outdoor environments using multisorbent samplers, and to subsequently analyze the concentration of VOCs, as selected by EPA. The procedure involves several steps including the assembly of the sampling apparatus, the sampling of the indoor and outdoor air, and the analysis of the samples collected.

VOC samples are collected indoors (at Fixed Sites 1, 3, and 5, as defined in the BASE Protocol) and at the outdoor site (Fixed Site 0), using diaphragm and peristaltic pumps to maintain flow rates of air to the multisorbent samplers.

# 2.0 GENERAL PROCEDURES

## 2.1 GENERAL SAMPLING CRITERIA AND REQUIREMENTS

The critical issues involved for proper collection of VOC samples involve the assurance of a constant flow rate over the sampling period, handling, storage and shipment of the sampler to prevent contamination, and the analytical procedures employed by the laboratory. Multisorbent samplers must be prepared to ensure a clean sampler. The air drawn through the sampler must be at a constant rate over the time period for which the integrated sample is required. The storage and transport of the canister must occur without loss or breakage of sample or contamination by extraneous materials. The speciation and analysis of selected compounds will be conducted by prescribed analytical procedures (gas chromatography and mass spectrometry), which generally follow the steps outlined in EPA Method TO-1.[1]

[1] Compendium of Methods for Determination of Air Pollutants in Indoor Air, EPA, 1989.

## 2.2 REQUIRED EQUIPMENT AND SUPPLIES

- 7 multisorbent samplers[1]
- 1 spiked multisorbent sampler[2]
- 1 field blank multisorbent sampler
- 3 diaphragm pumps with flow controller assemblies[2]
- 1 peristaltic pump[3]
- Two (2) medium size adjustable wrenches
- IADCS sample ID labels.
- Sample data sheet.

## 2.3 SAMPLING APPARATUS.

The multisorbent sampler consists of a 200 mm long by 6 mm O.D. glass tube packed with several sorbent materials, as detailed in Figure 1 of appendix A. There is a glass frit at the sample inlet end, followed by sections of sorbent materials consisting of: 1) glass beads; 2) Tenax TA; 3) Ambersorb XE-340; and 4) activated carbon. Each section is separated by glass-wool plugs.

When not in use, the sampler is capped at each end with a Swagelock nylon plug and nut fitted with Swagelock two-piece 6 mm Teflon ferrules. The caps should be sealed tightly enough to prevent slippage off the glass tube. For additional protection, the capped sampler is contained within a glass vial with a screw cap closure.

### Sampling Train

Figure 2 of appendix A details a schematic of the indoor sampling train and pump module. For indoor measurements, the sampling train downstream of the multisorbent samplers consists of a pump mode containing an Inlet Unit, a Control Unit, and a diaphragm pump.

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1 Part No. ST-032, Envirochem, Inc., Kemblesville, PA.

2 Supplied by Berkeley Analytical Associates, Richmond, CA.

3 Masterflex L/S by Cole-Parmer Instrument Co., Niles, IL.

The Inlet Unit allows for the simultaneous collection of duplicate samples using a single pump. Duplicate collection is advantageous in providing back-up or duplicate samples, and/or flexibility in the analytical strategy.

The Control Unit is connected with the Inlet Unit on the right using a U-shaped stainless steel tube. The bulkhead-union ports are located on the tops of the units and are indicated with the labels, “To Inlet Unit” and “To Control Unit.” The diaphragm pump is connected to the control unit using two pieces of polyethylene tubing. The inlet of the pump is connected to the union on the top of the Control Unit labeled, “To Pump Inlet.” The outlet of the pump is connected to the union labeled, “To Pump Outlet.” All swage fittings should be tightened with a wrench to ensure an adequate seal.

The Inlet Unit consists of paired rotameters used to balance the flow rates for the duplicate VOC samples. The rotameter on the Control Unit is used to set the total combined flow rate for the duplicate VOC samples. This is accomplished through the manipulation of a series of valves on the Control Unit.

While sampling, the pump module should be set on a foam mouse pad or cushion to reduce vibration noise.

The outdoor sampling apparatus consists of duplicate, multisorbent samplers attached to C-flex tubing. This tubing is required to accommodate the remote locations often associated with outdoor sampling. A peristaltic pump is used to maintain a constant flow rate over the desired time period. Due to the nature of the peristaltic pump, flow rates are constant and can not be adjusted.

## 2.4 PREPARING THE SAMPLERS

Prior to sampling, each sampler is labeled using an IADCS generated ID label. This label is affixed to the glass vial in which the sampler was enclosed. **IADCS ID labels must not be directly affixed to any portion of the sampler.** Each sampler also has a unique identifying number embossed or etched on the tube by the laboratory. These numbers are recorded on the sample log sheet.

The sampling apparatus are set up at their appropriate sites the evening before the sampling period. The media is deployed and sampling begins on Wednesday morning of the study week at approximately 07:30 and continues until approximately 17:30 on Wednesday afternoon. These sampling start and stop times are typical. However, they should be adjusted appropriately to capture the working hours of the study area occupants.

The sampling period begins once the pump is activated and the sampler is connected to the Inlet Unit. The time that air begins flowing through the sampler is recorded on the log sheet as "On Time." Also recorded on the log sheet is the IADCS ID number, the laboratory ID number, and the pump ID code.

To end the sampling period, the pump is unplugged. This time is recorded as "Off Time" on the log sheet. The samplers are removed from the Inlet Unit and the ends of the sampler are sealed using the Nylon nut and cap assemblies. The sampler is then placed inside the glass vial for protection during shipment.

## 2.5 INSTALLING THE SAMPLERS

For indoor sampling, multisorbent samplers can be attached directly to the Inlet Unit with the ¼ Swagelock union using the Nylon nut and 6 mm Teflon ferrules on the glass tube. The nut should be hand tightened so the sampler can not be pulled from the union. Each sampler is attached so that the tape label at the inlet end of the sampler is pointed away from the connection. The glass wool should be at the bottom of the sampler. See Figure 2 for a diagram of flow direction.

Flow rates can now be adjusted by measuring the total flow rate at the outlet of the Control Unit with a primary standard measuring air flow measuring device. Any flow rate calibration must be accomplished downstream of the samplers to prevent inadvertent contamination. Once the desired total air flow rate is set on the Control Unit, the paired rotameters controlling the individual flows to the samplers must be balanced. For example, if 5 cc/min is the desired flow rate for each of the samplers, a total flow rate of

10 cc/min should be set on the Control Unit. Provided the paired rotameters are balanced on the Inlet Unit, 5 cc/min will be equally distributed between each of the multisorbent samplers.

Sample flow rates may also be set on the Control and Inlet Units using “dummy” samplers to simulate the pressure drop across samplers. This method may be useful in situations where time is limited, however this procedure does not account for the varying pressure drop across individual samplers and may result in erroneous sample collection volumes.

Since the peristaltic pump does is not equipped for flow rate adjustments, it is prudent to use “dummy” samplers to determine the constant pump flow rate. As the peristaltic pump is able to overcome most pressure drops, the resultant error in collection volume should be minimal.

## 2.6 SET-UP AND SAMPLING

### Indoor Sampling

Before installing the samplers, the pump should be plugged in, and the On/Off valve should be set to the “On” position by rotating the valve to the horizontal position. The “adjust flow” valve on the Control Unit may be opened one turn, and the paired rotameters can be balanced to a reading of 60 on both rotameters. It may be necessary to increase the total flow with the “Adjust Flow” valve to obtain this reading. These settings are approximate and will have to be re-adjusted after the samplers are installed and the system is operating.

## 2.7 SAMPLE VOLUME AND FLOW RATES

The correct sample volume depends upon the concentrations of VOCs in the space being sampled. The analytical system has exceptional sensitivity, but limited range. For most investigations conducted in office buildings, the optimal sample volume is 2-3 L. A similar volume can be used for outdoor samples. This volume produces limits of detection for many individual VOCs that are typically about 1 $\mu g/m^3$.

For an integrated concentration of VOCs over an eight hour work period, a sample flow rate of 5 cc/min results in a sample volume of 2.4 L. The outdoor peristaltic pump runs at a constant flow rate of approximately 8.5 cc/min for an estimated 8-hour collection volume of 3.8 L.

## 2.8 SAMPLING LOCATIONS AND PRECAUTIONS

The fixed site sampling convention is as follows provided the study area can accommodate the configuration.

- Outdoor Site: One sample, one duplicate
- Fixed Site 1 (indoors): One sample, one field blank[1]
- Fixed Site 3 (indoors): One sample, one spiked sample
- Fixed Site 5 (indoors): One sample, one duplicate[2]

Indoor samples are collected at a height of 1.1 meters. Outdoor samples are collected under a protective shelter to ensure that neither precipitation nor direct sunlight affect the samplers. It is not recommended that outdoor sampling be conducted in very high humidity situations, as the sampler will collect excess water vapor which may result in a loss of sample during GC/MS analysis. If these environmental conditions are unavoidable, the chain-of-custody form must indicate the humidity conditions so the lab

[1] Field blanks may be collected at either site F1, F3, or F5.

[2] Indoor duplicate samples may be collected at either site F1, F3, or F5 and may be placed based on site physical restrictions. Indoor duplicate samples shall not be collected across multiple fixed indoor sites (e.g., VOC duplicates at F1, particles at F3, and other duplicate samplers at F5).

can take steps to eliminate the excess moisture prior to analysis. Sampling is not recommended at locations with high ozone concentrations. Excessive ambient ozone concentrations may oxidize some sample components and degrade the Tenax sorbent material resulting in the production of artifacts.

# 3.0 QUALITY CONTROL

## 3.1 QC SAMPLES

One field blank will be submitted for laboratory analysis on a per building basis as a blinded sample, to ensure the quality of contaminant-free samplers. Additionally, duplicate samples will also be submitted to assess the repeatability (precision) of this analytical method. Each week one set of duplicate samples is collected at a specified indoor location and another set of duplicate samples is collected at the outdoor location.

To test the accuracy of the analytical procedures, samplers will be spiked for subsequent analysis. Samples are spiked at the laboratory and delivered to the field team for each city as part of the normal sample shipment. These spiked samplers are issued an IADCS generated ID label disguised with all other VOC samples and returned to the laboratory for analysis.

## 4.0 SHIPPING AND HANDLING

A fresh lot of multisorbent samplers (including the spiked sampler) must be ordered for each week of sampling to reduce and standardize storage times. Leftover samplers can be used for sampling the following week, provided samplers can be transported using ice packs.

Samplers can be handled with ungloved hands, however care must be taken to avoid contamination. Hands should be free of dirt and oil before handling the media. Samplers should be held by the middle of the tube to avoid the ends near the openings.

The samplers should be stored, transported, and shipped within the tightly sealed glass vial. It is recommended that samplers be transported in ice chests with ice packs to keep the samplers cool. Situations that subject the samplers to elevated temperatures, such as might occur in closed vehicles parked in the sun, should be avoided. Prior to the use of samplers in the field, media should be stored in a freezer or refrigerator that is not used for the storage of chemicals. Under these conditions, the maximum recommended storage time is three weeks.

During storage, care must be taken to isolate the samplers from any DNPH (2,4-dinitrophenyl-hydrazine) samplers that have been used for the collection of formaldehyde and other carbonyl compounds. These DNPH samplers can be a serious source of acetonitrile contamination.

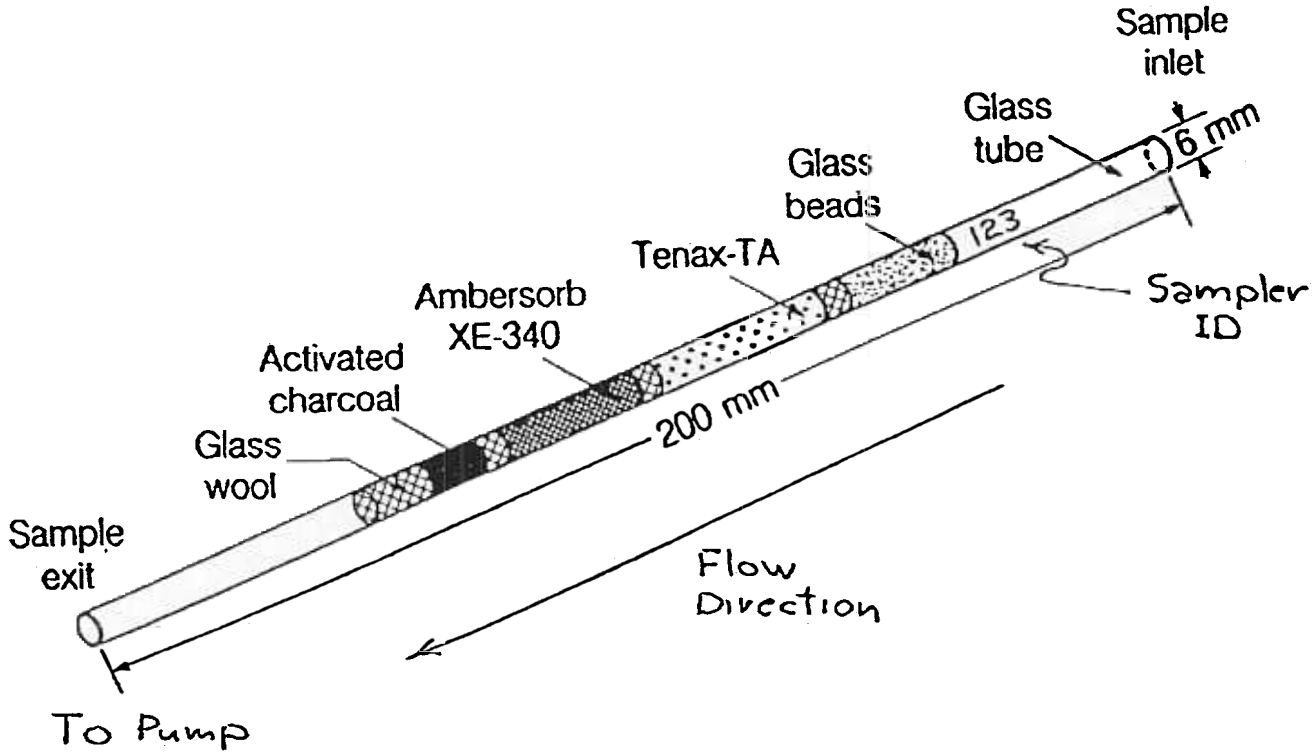
## 5.0 CHEMICAL ANALYSIS

The contaminants collected on the sample media are eluted by thermal desorption. The sample is then analyzed using high resolution gas chromatography/mass spectrometry by the methods outlined in Indoor Air Compendium, Method TO-1.

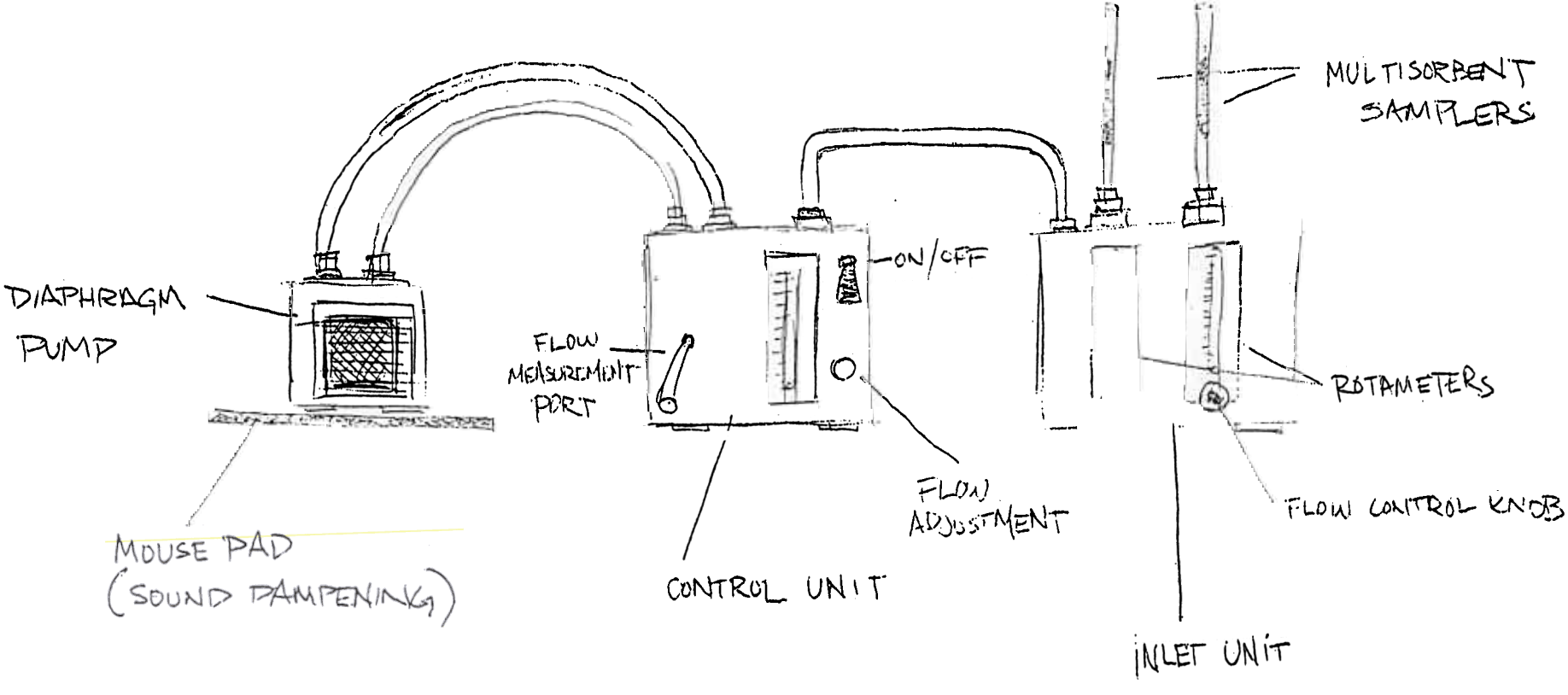
# APPENDIX A

**Figure 1**

**Multisorbent Sampler for Volatile Organic Compounds**



# FIGURE 2


MULTISORBENT SAMPLERS
ON/OFF
DIAPHRAGM PUMP
FLOW MEASUREMENT PORT
ROTAMETERS
FLOW ADJUSTMENT
FLOW CONTROL KNOB
MOUSE PAD (SOUND DAMPENING)
CONTROL UNIT
INLET UNIT