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Collection of Samples for Analysis of Volatile Organic Compounds (VOCs) in Indoor Air Using Sorbent Tubes

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Introduction

This guide is intended for indoor air quality (IAQ) investigations involving the sampling of volatile organic compounds (VOCs) onto sorbent tubes for subsequent laboratory analysis of individual and total VOCs (TVOC) by thermal desorption GC/MS. Determining the appropriate sampling strategy is a critical first step to achieving the study's objectives. Additionally, field air samples must be collected properly to obtain meaningful results. Specific procedures covered in this guide include selection of sample volumes, control and monitoring of the sampling process, reducing sources of VOC contamination, and sampler storage and transport. Multisorbent samplers containing Tenax™ TA backed up by a carbon molecular sieve are recommended for most IAQ projects. The use of multisorbent samplers for air sampling of VOCs is covered in U.S. EPA Method TO-17 (see **BAA** website for a link to this method).

Sampling Strategy

1. For many typical building investigations, it is recommended that samples be collected from several indoor locations and one outdoor location. As an example, if you are investigating a complaint building, you may consider collecting samples from the areas in the building with the highest complaint levels and at least one sample from an area in the building with a low complaint level to serve as a control. An outdoor sample may be collected to serve as a background reference. The outdoor location should be carefully selected to be representative of the ventilation air entering the building. For example, avoid hanging samplers immediately outside of open windows since air may be flowing out of the building at the windows.
2. It is recommended that two samples be collected at each location. These duplicate samples ideally should be co-located and collected simultaneously. This strategy provides backup samples in case there is a problem during analysis; and if both samples from a location are analyzed, a measure of sampling and analysis precision is obtained.

3. It may be desirable to have a field blank in addition to an outdoor reference sample. Often only one blank sample is needed per building. The blank sampler should be handled, transported and stored identically to the other samplers.

Description of the Sampler

1. The multisorbent sampler (Custom order, Supelco) for VOCs consists of a 3.5-in (90-mm) long by ¼-in (6.4-mm) passivated stainless steel tube packed with two sorbent materials (Figure 1). At the inlet, there is a section of Tenax™ TA (60/80 mesh), ~125 mg; this is backed up with a section of Carbosieve S-III (60/80 mesh), ~100 mg. There are two engraved rings at the inlet end. The sampler is installed so that the air being sampled enters the end of the tube closest to these rings. Each sampler has a unique identification number (alpha-numeric) engraved on the body of the sampler. This number should be used to identify the sampler in logs and on the chain-of-custody (COC) form.
2. The sampler is contained in a transparent acrylic tube with white plastic end caps fitted with Teflon-lined septa (Figure 1). The tube is slightly shorter than the length of the sorbent tube so the caps form positive seals at the inlet and outlet of the sampler. The caps should be hand tightened so the caps make firm seals but not over tightened to the point of damaging the septa. Do **not** use a wrench to tighten the caps.

Handling, Storage and Transport

1. Samplers typically can be handled with ungloved hands. However, care must be taken to avoid contamination. For example, if your hands are dirty, oily or treated with hand lotion, wash them and/or use gloves. Clean white cotton or nylon gloves are recommended. Hold the samplers by the middle and try to minimize touching the ends near the openings.
2. Do **not** attach labels directly to the samplers as these labels are a source of contamination. Also, do **not** write on the acrylic tubes with marking pens as these tubes are reused. If it is necessary to put labels on the outside of the acrylic tubes, please be sure these are easy to remove and do not leave an adhesive residue.
3. Store, transport and ship samplers tightly enclosed in their storage containers. Avoid subjecting the samplers to elevated temperatures such as might occur in closed vehicles parked in the sun. It is recommended that samplers be transported and shipped in an ice chest using ice packs to keep the samplers cool. Prior to use of the samplers in the field, store them in a freezer or a refrigerator that is not used for the storage of chemicals. Under these conditions, the maximum recommended storage time for unused samplers is approximately three weeks. Return the samplers to **BAA** for analysis as soon as possible after the collection of samples. If used samplers must be stored prior to shipment to **BAA**, keep them in a clean refrigerator or freezer.
4. When storing, transporting and shipping multisorbent samplers, make sure that they are well isolated from any DNPH samplers that have been used for the collection of formaldehyde and other carbonyl compounds. These DNPH samplers can be a source of acetonitrile contamination. At a minimum, it is recommended that capped and enclosed multisorbent samplers and used DNPH samplers be contained in separate, sealed polyethylene bags.

5. When submitting samplers to **BAA** for analysis, be sure to clearly indicate what services are desired. An IAQ services COC form is available for download on the **BAA** website if you do not already have an appropriate form.

Sampling Apparatus

1. If possible, draw air directly into a sampler so as to avoid connecting tubing to the inlet of the sampler. The only types of tubing that are acceptable for use upstream of the sampler are Teflon and stainless steel. Prior to use, any inlet tubing should be cleaned by flushing it with methanol and then thoroughly dried. Use a clean Swagelok ¼-in stainless-steel or Teflon union with ¼-in Teflon ferrules to join the inlet tubing to the sampler. Please do **not** use metal ferrules.
2. The sampling pump must be an oil-less type. There are a variety of AC or DC voltage diaphragm and peristaltic pumps that are suitable. It is advisable to check the compatibility of your pump using a spare sampler(s) prior to the collection of field samples. Fixed-speed peristaltic pumps are available on a rental basis from **BAA**.
3. The sampler must always be connected upstream of the sample pump. In some cases, the sampler can be connected directly to the inlet of the pump. Often it is desirable to locate the sampler away from the pump and connect the sampler to the pump with tubing. Inexpensive and easy-to-handle sample tubing is 1/4" O.D. polyethylene tubing. Other acceptable types of tubing are Teflon, Viton, silicon rubber, food-grade rubber, stainless steel and copper. Tubing composed of soft polymeric materials, such as Tygon, should be avoided. Use a clean Swagelok ¼-in union fitted with ¼-in Teflon ferrules to join the tubing to the outlet of the sampler. Please do **not** use metal ferrules. Alternately, flexible tubing (e.g., Viton and silicon rubber) sized appropriately to create a good seal can be slipped over the outlet of the sampler.
4. Aluminum, U-shaped brackets for mounting samplers may be available from **BAA** or can be easily fabricated. In office buildings, these brackets can be mounted to metal surfaces such as the tops or sides of filing cabinets and bookshelves with blue masking tape (avoid using tape on easily damaged surfaces).

Sample Volume and Flow Rate

1. It is imperative to know the flow rate of air through the sampler and the sampling interval elapsed time in order to calculate the sample volume. For highest precision, the flow rate through the sampler should be maintained at a constant rate and monitored continuously, or at least frequently, during sampling. These objectives are best accomplished using electronic mass flow controllers in combination with a vacuum pump. Alternately, a pump with integrated flow control or a separate needle valve may be used to maintain constant flow rate. Electronic flow sensors, positive displacement flow sensors, bubble flow meters, or a rotameter may be used to monitor the flow rate.
2. The correct sample volume depends upon the concentrations of VOCs in the space being sampled. The analytical system has exceptional sensitivity, but limited range. Therefore, it is important to avoid collecting a sample volume that is too large and that may result in

overloading of the GC/MS system and in the loss of data for the sample. For most investigations conducted in office buildings or homes, the optimal sample volume is 5 - 6 L, or less. The same volume can be used for outdoor samples. This volume produces limits of detection for many individual VOCs that are typically about $1 \mu\text{g m}^{-3}$, or $<0.5 \text{ ppb}$. If there is reason to suspect that the concentration exceeds several mg m^{-3} as TVOC, then a correspondingly smaller sample volume should be used. Examples are sampling locations with recent use of solvent-containing products or with strong odors. In situations in which there is uncertainty about the concentration, at least two samples of different volumes should be collected. For example, volumes of 1 and 5 L typically would cover the situation in which the concentration of TVOC could range up to about 10 mg m^{-3} . In specialized cases, a much lower volume may be most appropriate. Please consult with **BAA** if you have any questions regarding the selection of a sample volume.

3. Every effort should be made to select the correct sample volume. However, an advantage of collecting duplicate samples is that the analytical split ratio can be changed if the analysis of the first sample overloads the GC/MS system. The standard split ratio used for analysis is 2:1. This ratio can be changed to 5:1, or higher if needed; but, large split ratios may introduce more uncertainty into the quantitative results for the sample.
4. The sample flow rate can range from a low of about $10 \text{ cm}^3 \text{ min}^{-1}$ up to about $200 \text{ cm}^3 \text{ min}^{-1}$. If it is desired to measure the integrated concentration of VOCs over an eight-hour work day, a sample flow rate of $10 \text{ cm}^3 \text{ min}^{-1}$ results in a 4.8-L sample volume. A sample collected at $100 \text{ cm}^3 \text{ min}^{-1}$ for 60 min results in 6-L sample volume, also in the acceptable range for typical building investigations. The maximum flow rate is limited by the pressure drop across the sampler and the capacity of the sampling pump.
5. The sampling system should be leak checked prior to use, preferably with the sampler in place. With some types of pumps, this can be accomplished by capping the inlet of the sampler and checking that the flow rate at the outlet of the pump drops to zero.
6. The air flow rate through the sampler must be determined. For flow regulation schemes other than those using electronic mass-flow controllers, it is necessary to directly measure the flow rate. At a minimum, this measurement should be made both near the beginning and end of the sampling interval. If possible, the measurement should be made downstream of the sampler, preferably at the outlet of the pump. A bubble flow meter, calibrated rotameter or other flow measurement device may be used. Record the measurement data including the air temperature and the atmospheric pressure so that the flow rate can be converted to standard indoor conditions (*i.e.*, 25° C , 1 atm). If the measurement must be made upstream of the sampler, briefly use a calibrated rotameter following the precautions described above. Do **not**, under any circumstance, attach a bubble flow meter upstream of the sampler. This will contaminate the sampler. Also note that BIOS International DryCal Flow Calibrator systems are a potential source of methyl methacrylate contamination when used upstream of a sampler.

Other Sampling Precautions

1. Do not expose samplers used outdoors to direct sunlight. Aluminum foil can be used to make a simple sun shield.
2. If possible, avoid collecting samples in the rain or in very high humidity situations. The sampler will collect excess water vapor, which may result in the loss of the sample during GC/MS analysis. If such a sampling situation is unavoidable, be sure to indicate the humidity conditions on the sample COC form as excess humidity can be removed in the laboratory by purging with dry helium prior to an analysis.
3. If possible, avoid collecting samples at locations with high ozone concentrations as ozone may oxidize some sample components and can degrade the Tenax sorbent material resulting in the production of artifacts. The use of an ozone scrubber generally is not recommended as sample components can be lost by sorption to the scrubber.

Figure 1. Multisorbent Sampler for VOCs

