

National Human Exposure Assessment Survey (NHEXAS)

Arizona Study

Quality Systems and Implementation Plan for Human Exposure Assessment

The University of Arizona
Tucson, Arizona 85721

Cooperative Agreement CR 821560

Standard Operating Procedure

SOP-BCO-L-18.1

Title: Preparation of Multisorbent Tubes for Actively-Pumped VOC Samplers

Source: The University of Arizona

U.S. Environmental Protection Agency
Office of Research and Development
Human Exposure & Atmospheric Sciences Division
Human Exposure Research Branch

Notice: The U.S. Environmental Protection Agency (EPA), through its Office of Research and Development (ORD), partially funded and collaborated in the research described here. This protocol is part of the Quality Systems Implementation Plan (QSIP) that was reviewed by the EPA and approved for use in this demonstration/scoping study. Mention of trade names or commercial products does not constitute endorsement or recommendation by EPA for use.

Preparation of Multisorbent Tubes for Actively-Pumped VOC Samplers

1.0 Purpose and Applicability

This standard operating procedure (SOP) describes the procedures followed in the preparation of carbon-based multisorbent tubes for the collection of volatile organic compounds (VOCs) in air using actively-pumped samplers.

2.0 Definitions

- 2.1 Multi-bed sorbent: two-phase or three-phase carbon-based sorbent packing materials used to trap $C_2 - C_4$ VOCs or $C_3 - C_{12}$ VOCs, respectively, in air.
- 2.2 Active sampler: tube packed with carbon-based sorbents used to collect air samples for organic vapor analysis by drawing air at a known flow rate through the tube using a pump.
- 2.3 Thermal desorption unit: device used to strip VOCs from sorbent material by passing helium gas through the cartridge while it is being heated.

3.0 References

- 3.1 "Carbotrap™ 300 Multi-Bed Thermal Desorption Tubes," Data Sheet, Supelco, Bellefonte, PA, 1992.
- 3.2 "Carbotrap™ 200 Multi-Bed Thermal Desorption Tubes," Data Sheet, Supelco, Bellefonte, PA, 1992.
- 3.3 Hewlett Packard 5880 GC Operator's Manual, ND; Hewlett Packard 5970 Mass Selective Detector Hardware Manual, ND.

4.0 Discussion

- 4.1 Nominally clean commercially-available Carbotrap 200 and 300 Thermal Desorption Tubes are conditioned in the laboratory before use, to ensure acceptable background levels of VOCs and consistent tube-to-tube performance.
- 4.2 Before use, each tube is subjected to thermal desorption in batches of ten tubes at a time. One of the tubes is selected at random and analyzed to ensure the integrity

of the cleanup and desorption procedure, and the conditioned tubes are packed and stored until needed.

5.0 Responsibilities

- 5.1 Battelle will be responsible for purchasing the multisorbent tubes and for conditioning them.
- 5.2 Battelle will check for background contamination in the cleaned sample lots of tubes prior to shipment of the materials to UA. Representative tubes will be thermally desorbed and analyzed per SOPs BCO-L-31.0 and BCO-L-22.1 to determine residual VOC levels in Carbotrap 200 and Carbotrap 300 tubes, respectively.
- 5.3 Battelle will ship the multisorbent tubes to UA.

6.0 Materials and Reagents

6.1 Materials

- 6.1.1 Carbotrap 300 Stainless Steel Multi-Bed Thermal Desorption Tubes (Supelco, Catalog No. 2-0370), containing 20/40 mesh Carbotrap C (300 mg), 20/40 mesh Carbotrap B (200 mg), and 60/80 mesh Carbosieve S-III (125 mg), in ¼" o.d. x 7" (18 cm) stainless steel.
- 6.1.2 Carbotrap 200 Stainless Steel Multi-Bed Thermal Desorption Tubes, containing 70/80 mesh glass beads, 20/40 mesh Carbotrap B, and 60/80 mesh Carbosieve S-III, in ¼" o.d. x 7" (18 cm) stainless steel.
- 6.1.3 Varian 3700 Gas Chromatograph (GC) oven.
- 6.1.4 Polyethylene sample bag with zipper lock.
- 6.1.5 Helium carrier gas (purity \geq 99.995%).

7.0 Procedure

7.1 Conditioning the Sampler Tubes

- 7.1.1 Select a batch of Carbotrap 200 and Carbotrap 300 tubes, as required, for conditioning.

- 7.1.2 Remove the end caps from the tubes, and mount the tubes on the manifold inside the GC oven. The manifold can hold up to 10 tubes at a time.
- 7.1.3 Condition the tubes in the GC oven by heating to 340°C under a helium purge of 100 mL/min for about 2 h.
- 7.1.4 At the end of the conditioning period, shut off the GC heater and allow the unit to cool to room temperature while the helium purge flow is maintained at 100 mL/min.
- 7.1.5 Remove each tube and replace the two end caps to seal the tube.
- 7.1.6 Place the tubes in polyethylene bags with zipper lock, and label the bags with desorption date and lot number of the tubes.
- 7.1.7 Store in refrigerator at 4°C until shipment to UA.
- 7.1.8 Record the lot number and the date on which the conditioning was carried out in the Multisorbent Tube notebook (see Section 8.0).
- 7.1.9 Storage for more than 1-2 weeks should be avoided.

7.2 Acceptance Test

See Section 7.4 for details.

7.3 Calculations

See Section 7.4 and SOPs BCO-L-31.0 and BCO-L-22.1 for further details.

7.4 Quality Control

7.4.1 Laboratory Blanks/Material Checks

- 7.4.1.1 A background check is run on ~10% of the tubes selected at random from batches of conditioned tubes.
- 7.4.1.2 Analyze one Carbotrap 200 and one Carbotrap 300 tube (if both types have been cleaned) using a Hewlett Packard GC/MSD equipped with a thermal desorption injection unit and interfaced to a data system (see SOPs BCO-L-31.0 and BCO-L-22.1 for details). Blank samplers should show peaks that are no greater than 5 ng per target compound. In practice, clean sorbent tubes

generally exhibit background peaks that are below the detection limit. (The method detection limit is about 0.3 ppbv. This is equivalent to the injection of a 5 ng sample collected from the passage of a 4 L air sample through a sorbent tube, and assuming a molecular weight of 100 for a typical target VOC.)

7.4.1.3 Record the measured peak areas as well as their sum in the laboratory notebook, and compare with the historical values corresponding to the limit of detection (LOD) for each target compound. The historical data are obtained prior to the tube acceptability tests by loading each target compound at the GC/MS expected LOD and determining its area under the same operating conditions by GC/MS analysis.

7.4.1.4 If the peak areas of any of the target compounds are equal to or exceed the LOD, the set of tubes is rejected and must be desorbed again. If the peak areas of any nontarget compounds are greater than five times the LOD, the set of tubes is rejected and must be desorbed again.

7.4.2 Overall Tube Performance

7.4.2.1 The overall performance of the sorbent tubes for monitoring VOCs in air is evaluated using spiked controls, blanks, and duplicates.

7.4.2.2 At least one sampler should be prepared for analysis as a field spike, one sampler each presented for analysis as a field blank and an unexposed blank, and one field duplicate sampler taken with every 30 field samples.

8.0 Records

8.1 The laboratory notebook assigned to the project for multisorbent tubes conditioning will contain the record of all relevant information for the tubes.

8.2 This record book will contain for each batch cleaned the batch number, the date of desorption, the purchase order number for the tubes and the date received.

8.3 This record book will also contain the target VOC background levels in the pre-shipment blanks.

- 8.4 This record book will be retained in the laboratory where these operations are performed and will be transferred to the office of the Battelle co-PI at the conclusion of the program.