U.S. EPA BASE STUDY
STANDARD OPERATING PROCEDURE FOR SAMPLING ALDEHYDES IN INDOOR AIR USING A SOLID ADSORBENT

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1.0 OBJECTIVE

The objective of the procedure described is to collect on beds of solid adsorbent (silica gel coated with 2,4-dinitrophenyl hydrazine [DNPH]) representative samples of formaldehyde and other carbonyl compounds present in an indoor environment and in the outdoor air supplied to the space, and to subsequently quantitative the amount collected, by desorption and chromatographic analysis. The test is intended to characterize the indoor level of aldehydes and its likely source (indoor or outdoor).

The procedure involves several steps generally performed by two or more groups of technical personnel. The critical issues in the process are as follows:

1. Preparation of a clean adsorbate, adequately encapsulated to be fully protected from contamination prior to exposure to the environment to be tested.

2. Exposure of the adsorbent to a controlled volume of the air to be tested, under conditions that preclude contamination of the environment by any materials derived from the test procedure itself.

3. Storage and transport of the laden adsorbent to prevent loss of adsorbed formaldehyde or contamination with extraneous materials.

4. Thermal desorption of the adsorbate and its analysis for selected compounds by prescribed analytical procedures (high performance liquid chromatography - HPLC, Ambient Air Compendium Method TO-11).

2.0 GENERAL PROCEDURES

2.1 SAMPLING CRITERIA AND REQUIREMENTS

The accurate sampling of the air to be tested depends on drawing a metered amount of air through the adsorbent containing tube. This is accomplished by drawing air through the adsorbing (Sep-Pak) cartridge at a constant rate for a measured time span with an air pump. The time span will be selected to cover the interval over which the integrated aldehyde concentration is of interest (the time of occupancy of the space studied). The total volume sampled depends also on the rate at which air is passed through the sampler, and the length of the sampling time interval. The flow rate will be adjusted to allow detection of concentrations of interest. The BASE Protocol specifies that 96 liters (±5%) of ambient air be passed through the standard Sep-Pak cartridge at a uniform flow rate of 200±10 ml/min during a continuous eight hour period.

2.2 REQUIRED EQUIPMENT AND SUPPLIES

- Sep-Pak[1] cartridges (all must be from the same manufacturer sample lot)
- "Dummy" cartridge (Sep-Pak used to check flow rates prior to sampling)
- Ozone scrubbers[2]
- Charcoal tubes[3]
- Personal sampling pumps
- Latex tubing
- Polyethylene tubing (for outdoor sampling only)
- Calibrated rotameter suitable for 200 cc/min flow rates.
- Uncoated steel paint cans
- Loose charcoal for shipping
- IADCS sample ID labels

[1] Manufactured and supplied by Millipore Corporation/Waters Chromatography Division, 34 Maple Street, Milford, MA 01757

[2] The ozone scrubber is made of a 36" long section of 1/8" ID copper tubing. A coating of potassium iodide (KI) is placed on the inside of the tubing. The tubing is coiled to a diameter of approximately 3" and the ends outfitted with proper fittings for connection into the sampling train.

[3] Typically an SKC #226-09 tube - approximately 4" long with a 1/4" ID, about 2/3 filled with granulated charcoal.

2.3 SAMPLING TRAIN DIAGRAM

The gas train consists of an ozone scrubber (potassium iodide-based denuder) at the front end, followed by the Sep-Pak cartridge. Immediately behind the cartridge is placed a trap filled with activated charcoal to remove acetonitrile liberated from the DNPH during sampling. The pump (low noise personal pump with a low flow adapter) is located downstream of the charcoal trap. The diagram shown in Figure 1 of Appendix A illustrates schematically the configuration of the sampling train.

The outdoor sampling train is identical, except for a length of polyethylene tubing connecting the outdoor sampling train to the pumps located in an indoor area for protection from the elements. (This may not be necessary for all outdoor sampling areas).

2.4 SET-UP AND SAMPLING

Accurate sampling of the air to be tested depends on drawing a metered amount of air through the cartridge. This is accomplished by drawing air through the cartridge at a constant rate for a measured time span. In order to insure a proper flow rate on the day of sampling, personal pumps are charged prior to sampling and are be briefly checked on the day before the scheduled sampling using a calibrated rotameter for a flow rate of 200±10 cc/min, and adjusted to it, if necessary. The rotameter reading check is conducted with the full sampling train in place and with a dummy cartridge replacing the actual sampling Sep-Pak.

Sampling trains are set up at their sites the night before sampling with a dummy cartridge in line to facilitate early start of sampling on the following morning. On the morning of the day of sampling, the personal pumps are warmed up for five minutes. Then a preliminary flow check is made with the pump connected to the sampling train and the dummy cartridge still in place. This is done by connecting the rotameter at the inlet of the sampling train and then measuring the flow rate. Once the 200±10 ml/min flow rate has been set on the pump based on the rotameter reading, the pump is turned off and the dummy cartridge replaced by the actual sampling Sep-Pak cartridge.

To set up the sample cartridge, the two Luer plugs on each end of the cartridge are removed and returned to the protective foil envelope, and the cartridge is inserted in the appropriate position in the sample train (replacing the dummy cartridge used for flow rate checking). Once the sample cartridge is in line, the pump is turned on and sampling started. Upon turning the pump on, the "Time On" for the sample is recorded. The flow must be rechecked with the Sep-Pak in line and readjusted to the rotameter reading yielding 200±10 ml/min, if necessary. The exact rotameter reading is recorded on the log sheet as the "Flow On" for the sample.

NOTE: Pressure drops across Sep-Pak cartridges are unpredictable. Therefore, testing flow rates with a dummy cartridge is insufficient. Such testing only serves to adjust to an approximate value. Final adjustment must be made with the actual sampling Sep-Pak in place.

Pump operation must be verified at each site throughout the sampling period. This is especially important at the outdoor site.

Prior to stopping the sampling, the "Flow Off" is recorded by following the same procedure as outlined for measuring "Flow On". After recording the rotameter reading ("Flow Off"), the pump is turned off and the Luer plugs are replaced on the cartridge (the time is recorded as "Time Off"). The cartridge is placed in the accompanying foil envelope and the envelope is sealed with laboratory tape.

2.5 SAMPLING LOCATIONS SCHEME AND QC SAMPLES

Each week, the following samples will be taken:

- Outdoor Site: One sample, one duplicate
- Fixed Site 1: One sample, one field blank[4]
- Fixed Site 3: One sample, one spiked sample
- Fixed Site 5: One sample, one duplicate[5]

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

[5] 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).

3.0 CALIBRATIONS AND QUALITY CONTROL

The air flow rates through the sampling Sep-Pak are measured with calibrated flow meters. The quality control (QC) of samples collected on Sep-Paks is conducted by means of lab blanks, lab spikes, field spikes, field blanks, duplicates, and performance evaluation and demonstration samples.

3.1 PUMPS

The procedure for setting the air flow rate with reference to a calibrated flowmeter (rotameter) is described in Section 2.4. Each gas train (ozone scrubber, sample cartridge, absorbent bed, and pump combination) is different, and on-site flow rate adjustment is conducted at the beginning of each sampling run. The flow rate for the sampling period is taken as the average of the flow measurements at the beginning ("Flow-On") and end ("Flow-Off") of the sampling period. The data will be flagged if the discrepancy between "Flow-On" and "Flow-Off" exceeds 10 ml/min. If the discrepancy exceeds 15 ml/min, data should not be used.

3.2 SOLID ADSORBENT CARTRIDGES

The adsorbent-containing cartridges or traps (e.g. Sep-Paks) must be purchased from the manufacturer and must be clean of any volatile organic contaminants, as documented by proper quality control procedures. The cartridges typically used (e.g. Sep-Paks) are 1.5 cm ID x 2 cm long polyethylene tubes with Luer type fittings (plugs) at each end. The cartridges must be examined upon receipt for physical damage that may have occurred during shipping or any other visible defect. Cartridges showing apparent defects must be rejected.

A fresh lot of cartridges will be ordered at the beginning of the study to ensure consistency among samples. All regular samples and QC samples will come from the same manufacturer sample lot. Thus the sampling cartridges to be used for each building study are ordered at the same time.

The quality control samples for the adsorbent cartridges for the BASE study will consist of lab blanks, lab spikes, a field blank, a field spike, and performance evaluation/demonstration samples.

The field blank is prepared by going through the process of placing a cartridge in line for 30 seconds and then removing it without having drawn air through the sample. Then the field blank is placed in a foil envelope to sit at the blank fixed site for the duration of the sampling period.

The laboratory blanks and spikes and the field spiked sample cartridges are prepared by the analytical laboratory. Approximately two lab blanks and lab spikes per city should be adequate. The field-spiked sample is sent by the laboratory to the field on a weekly basis and is kept at the spiked site inside a foil envelope for the duration of the sampling period.

Performance evaluation and performance demonstration samples are prepared by an independent group and submitted once or twice per study depending on the total number of field weeks.

4.0 SHIPPING AND HANDLING

The BASE ID labels will follow a chronological sequence from one lot to the next and will be affixed to the Sep-Paks outer protective foil shipping envelope prior to being sent to the laboratory for analysis.

The Sep-Pak cartridges are kept refrigerated (except when sampling) to increase their shelf life. Samples are transferred from site to site inside a cooler.

After collecting the samples from all sites, they are prepared for shipping. The sealed envelopes are placed in an uncoated steel paint can with a 1/4" layer of charcoal on the bottom and closed tightly before shipping. The paint cans are placed inside cushioned hard-sided shipping containers. This packing procedure reduces the possibility of contamination of samples during shipping.

The packed samples are shipped on the day of sampling by overnight delivery to the laboratory.

5.0 ANALYTICAL METHODS

Aldehyde and other carbonyl compounds desorbed from the solid adsorbent tubes will be analyzed by high performance liquid chromatography following the Ambient Air Compendium, Method TO-11 (1987). Currently results are reported for only formaldehyde and acetaldehyde.

APPENDIX A

FIGURE 1
ALDEHYDE SAMPLING TRAIN

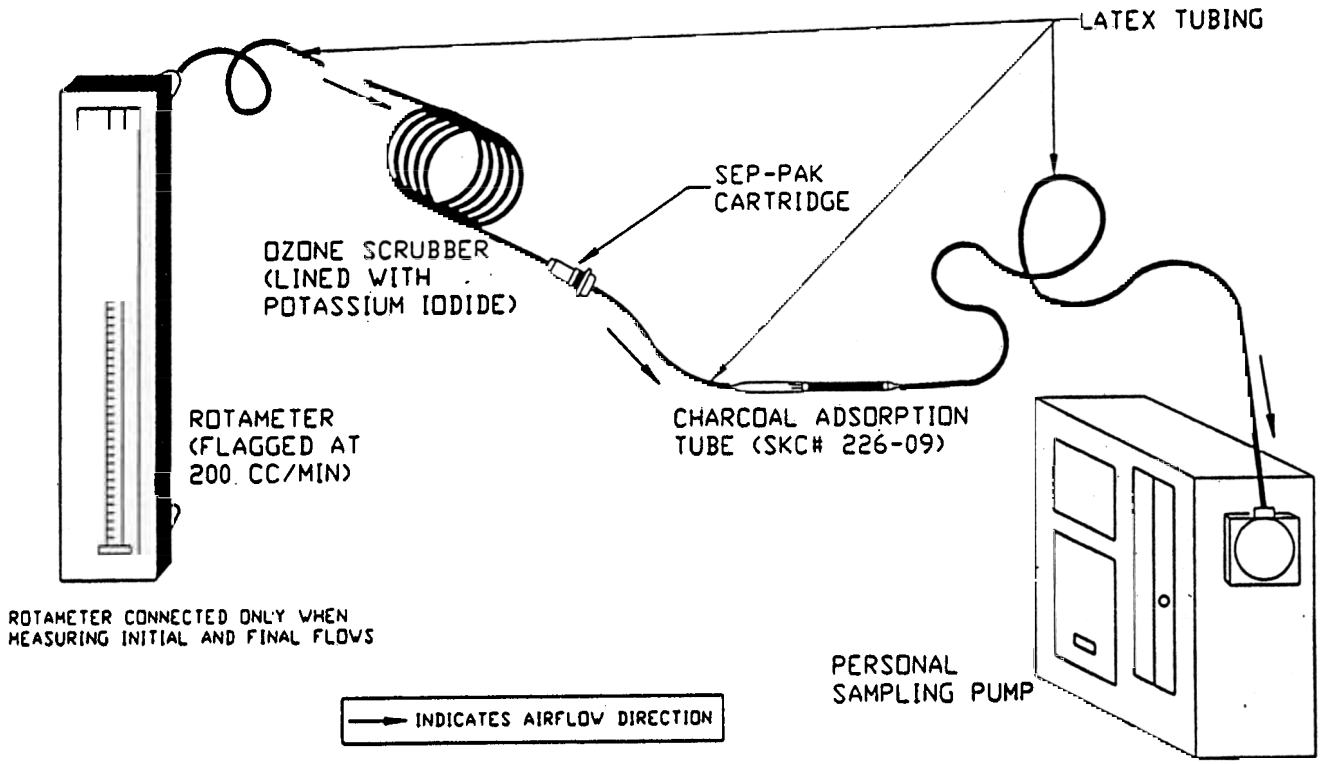

FIGURE 1 Aldehyde Sampling Train