

## Monitoring Airborne Contaminants in Workplace Atmospheres, Using Sampling Devices and GC or HPLC

*Workers' exposure to hazardous substances must be held within acceptable limits. Therefore, airborne contaminant vapors and particles (including aerosols) in industrial atmospheres must be monitored. In the United States, the National Institute for Occupational Safety and Health (NIOSH) and the Occupational Safety and Health Administration (OSHA) offer, for many such substances, specific recommendations on collecting (trapping) the contaminant on an adsorbent and/or filter, desorbing the collected analyte, analyzing the sample by gas or liquid chromatography, and quantifying the contaminant. Presented here is a general outline of the NIOSH and OSHA procedures, including formulas for calculating airborne concentrations of collected vaporous and particulate contaminants.*

### Key Words

- air sampling
- industrial hygiene

### Background

The National Institute for Occupational Safety and Health (NIOSH) and the Occupational Safety and Health Administration (OSHA) have published methods for monitoring many airborne contaminants. These methods are continually revised and updated as collection techniques improve and/or exposure limits change. NIOSH methods for monitoring approximately 500 airborne contaminants have been published in the fourth edition of the NIOSH Manual of Analytical Methods (1). OSHA has published similar methods for analyses of a smaller number of airborne contaminants.

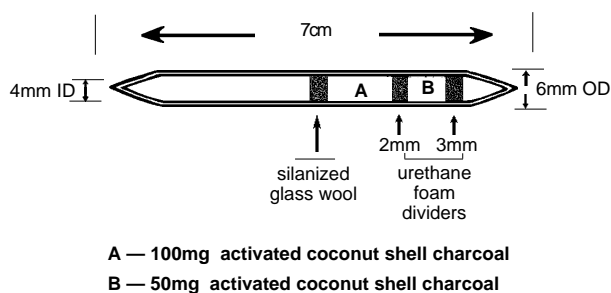
OSHA has restricted workers' exposure to the airborne contaminants listed in the appendix to this bulletin and in Reference 2. OSHA's permissible exposure limits (PELs) are expressed as time weighted average, short term exposure limit and ceiling values. The *time-weighted average* (TWA) is the maximum average concentration of an airborne contaminant to which a worker may be exposed for a normal eight-hour work day in a forty-hour work week. The *short-term exposure limit* (STEL) is the maximum concentration of an airborne contaminant that a worker can be exposed to continuously for a short period of time (15 minutes, unless specified otherwise). The *ceiling level* is the concentration of an airborne contaminant that should not be exceeded at any time during the work period.

### Collecting Airborne Contaminant Vapors

The recommended adsorbent tube, filter cassette, or other sampling device for collecting a specific airborne contaminant is described in the NIOSH or OSHA method for that analyte. For

example, in NIOSH Method 1400 an adsorbent tube containing 150mg of coconut charcoal, divided into a 100mg front bed and a 50mg backup bed (Figure A), is recommended for collecting alcohol vapors. Under the sampling conditions described in the method, the front bed efficiently traps the airborne analyte. The backup bed is monitored to ensure that no analyte has passed through the front bed. Such breakthrough would indicate possible sample loss and, therefore, would make the sample invalid. This two bed configuration is characteristic of most NIOSH-

**Figure A. Typical Adsorbent Tube for Collecting Airborne Compounds**



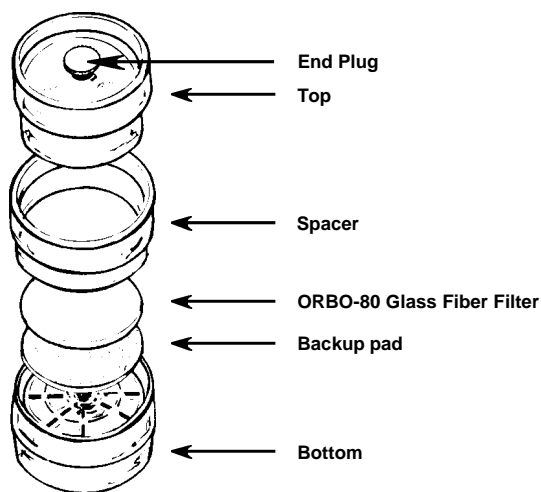
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recommended adsorbent tubes for collecting airborne contaminants. Some newer-design tubes incorporate both adsorbents and a filter (OSHA versatile sampler or OVS tubes). Other sampling methods call for using filter cassettes. A typical filter cassette is shown in Figure B.

To sample airborne vapors, break the sealed ends of the adsorbent tube or unplug the ends of the filter cassette and place the sampling device in a holder. Connect the holder to the inlet of a battery operated personal pump and adjust the pump to draw air through the tube at the appropriate flow rate (see appendix). The sampling device should be carried near the breathing zone of the person whose exposure is to be monitored. Alternatively, in area-sampling methods, simply place the sampling device in the area to be monitored. Air to be sampled should not pass through any other material (tubing, etc.) prior to entering the collection device. During sampling, keep adsorbent tubes in a vertical position to prevent shifts in the adsorbent bed that might allow vapors to channel through the bed.

Use the sampling rate and sample volume indicated in the method (see appendix), or the material to be monitored may not

**Figure B. Typical Filter Cassette for Monitoring Airborne Materials**



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be effectively trapped. If you wish to use another rate or volume, you must show that results are equivalent to those obtained by following the published method. Record temperature and atmospheric pressure at the sampling site, to correct the air sample volume to standard temperature (25°C) and pressure (760mm Hg). To establish a baseline (field blank) value, open and immediately cap a tube at the sampling site.

When the recommended volume of air has been drawn through the sampling device, remove the device from the holder and cap the ends. In some cases, adsorbent tubes can be refrigerated for analysis at a later date.

### Desorbing the Sample

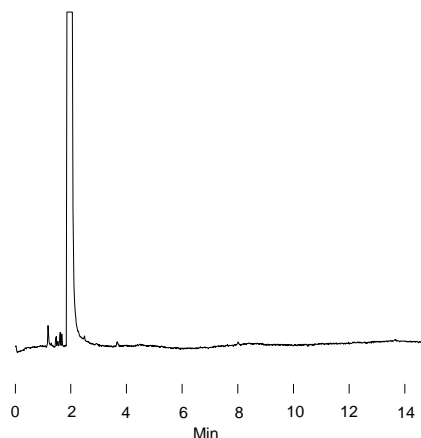
Desorb or extract the airborne contaminant from the adsorbent or filter, using the solvent recommended in the method, in a well-ventilated hood. During desorption, avoid exposing the samples to potential contaminants (airborne laboratory chemicals, finger oils, unclean vials, etc.). When sampling two or more analytes that require different desorbing solvents, NIOSH recommends obtaining separate samples for each analyte or using the solvent for the analyte with the lowest TWA value. Unused sampling devices (blanks) should be processed in the same manner as the devices containing samples.

Remove the adsorbent beds and, if appropriate, the retaining plugs and seal the front bed and the backup bed in separate vials. The vials typically recommended for desorption have a screw top with a hole in the center and a Teflon<sup>®</sup>-coated septum to permit sampling with a syringe.

Use only high-purity solvents (e.g., low-benzene CS<sub>2</sub>) for desorbing collected materials and analyze the desorbing solvent for impurities (Figure C). If necessary, subtract interfering peaks from peaks for sample components, or use an alternative lot of solvent. Inject the appropriate quantity of desorbing solvent through the septum of each sealed vial. Note the initial volume of the adsorbent and desorbing solvent in each vial by marking the bottom of the meniscus. This will enable you to monitor the liquid volume for decreases (volatilization) or increases (moisture uptake or expansion due to heat) that could affect your data.

**Figure C. Low-Benzene Carbon Disulfide Has Minimal Impurities**

Column: **SPB-5, 30m x 0.53mm, 1.5µm film**  
Cat. No.: **25305**  
Oven: 40°C (4 min) to 150°C at 6°C/min  
Carrier: helium, 8mL/min  
Det.: FID (300°C)  
Inj.: 1µL, direct injection, packed injector (220°C)



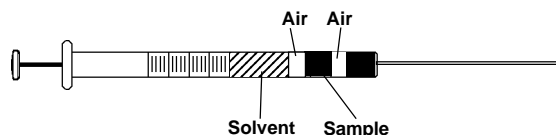
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Agitate the sample in an agitator or ultrasonic bath, for the length of time specified in the method, to completely desorb the analyte. If you cannot analyze the desorbed samples immediately, refrigerate them. Monitor for changes in analyte concentration by storing freshly prepared standards under the same conditions and analyzing them along with the samples.

### Analyzing Desorbed Samples

Following sample agitation, remove appropriate aliquots for chromatographic analysis by piercing the septum with a syringe needle. Do not pull any of the adsorbent from the bottom of the vial into the needle. Because reproducibility is critical, use the solvent flush technique of sample injection (Figure D). Make triplicate injections for each sample and average the peak areas for each compound of interest. An internal standard in the desorbing solvent (one that will not interfere with the desorption or chromatography) will also increase accuracy of the analysis.

**Figure D. For Reproducible Sample Injections, Use the Solvent Flush Technique**



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When using adsorbent tubes, analyze the adsorbent in the backup bed of each tube in the same manner as the front bed. Typically, if the total amount of the sampled compound in the backup section is greater than 10% of the total amount in the front section, sample may have been lost due to breakthrough. Consider the sampling results invalid.

Analyze samples trapped on filter cassettes as called for in the method you are following. Analyses can be by chromatography, gravimetry, or other techniques.

## Typical Chromatographic Analyses

NIOSH and OSHA methods typically list a GC column or HPLC column for analyzing the trapped analyte. In older methods, the GC column is often a packed column, but capillary columns will give better sensitivity, and thus more accurate results. Wide bore capillary columns — 0.53mm and 0.75mm ID — offer the additional advantage of usually being compatible with packed column GC equipment. Thus, you have the advantage of increased sensitivity without much additional expense for equipment.

### Solvents (GC)

Most common solvents, even in mixtures, can be analyzed on a bonded polyethylene glycol-type phase (e.g., SUPELCOWAX™ 10) or a methyl:5% phenyl polysiloxane phase (e.g., SPB™-5 or PTE™-5) capillary column. Figure E shows a separation of 50 common solvents on a 5% phenyl polysiloxane phase column.

Analyses of alcohols and other extremely polar compounds are a problem, because peaks for these active analytes can tail or be poorly resolved. For these analyses, we suggest a SUPELCOWAX 10 or SPB-1701 (bonded OV®-1701 vinyl silicone phase) capillary column (Figure F). Either of these columns will ensure good peak symmetry for polar compounds. For more information about analyzing solvents on capillary columns and packed columns, request **Bulletin 824** and see the applications section of our general catalog.

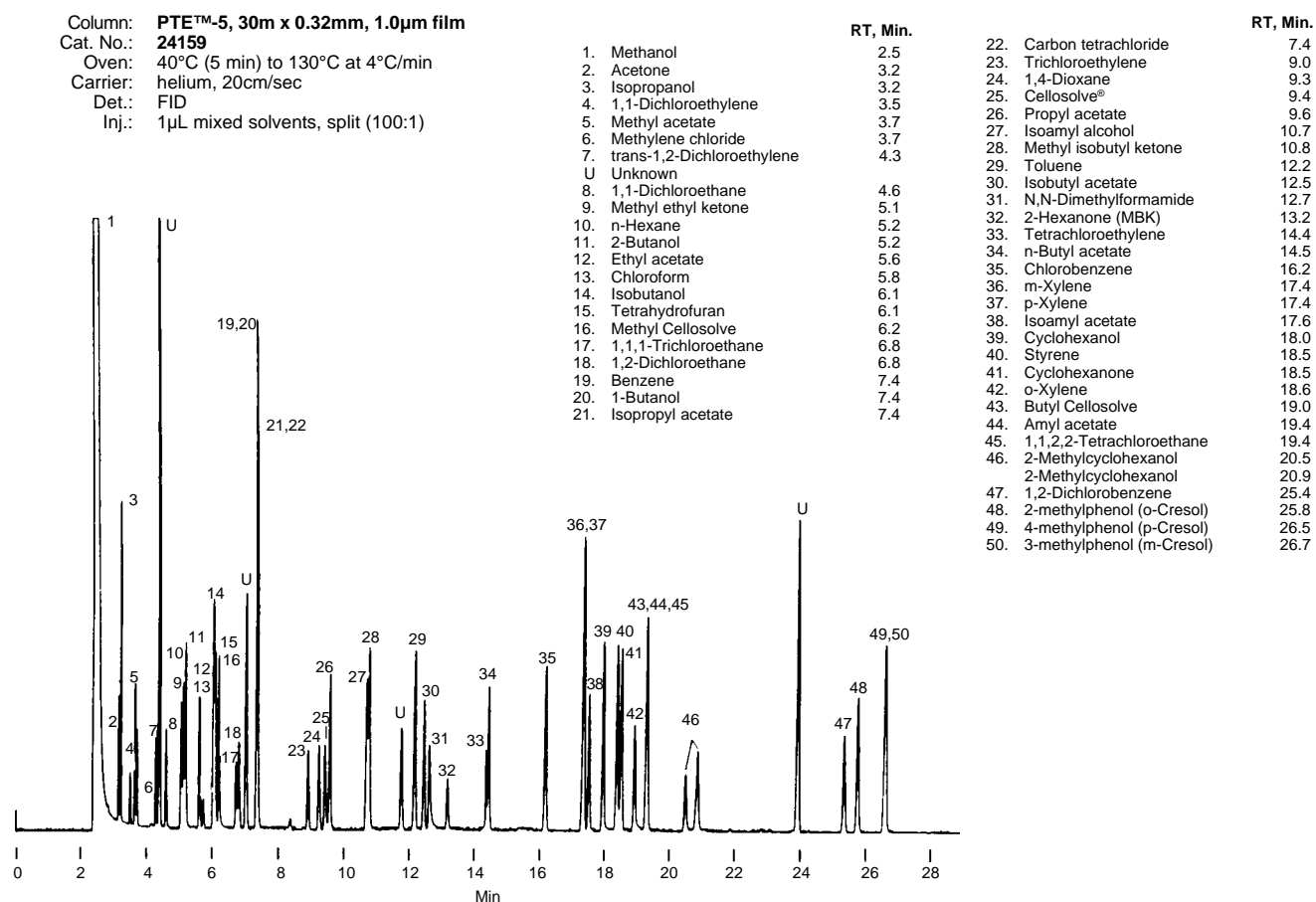
### Pesticides (GC)

Airborne pesticides can be collected on ORBO™-49P adsorbent tubes (Supelpak™-20P resin, glass fiber filter) or ORBO-1000 polyurethane foam plug tubes, as specified in OSHA and American Society for Testing and Materials methods, respectively, then analyzed by capillary GC (Figures G and H). Low background in these ORBO tubes generally enables the analyst to monitor pesticides at well below the limits specified in these methods.

### Inorganic Acid Anions (IC)

Common inorganic acid anions ( $\text{Br}^-$ ,  $\text{Cl}^-$ ,  $\text{NO}_3^-$ ,  $\text{PO}_4^{3-}$ ,  $\text{SO}_4^{2-}$ ) can be trapped on ORBO-53 adsorbent tubes (silica gel adsorbent). These tubes incorporate a glass fiber filter, as described in NIOSH Method 7903, to trap acid anions in aerosol form. A suppressor column/conductivity detector ion chromatography analysis is described in the NIOSH method. Quantification is best performed using weight/volume concentrations (similar to the calculations described under "Quantification" on the following pages). Figure I shows the anions can be monitored at very low levels. Suppressed ion chromatography has also provided good results in analyses of  $\text{Cl}^-$ ,  $\text{NO}_3^-$ , and  $\text{SO}_4^{2-}$  in snow (3).

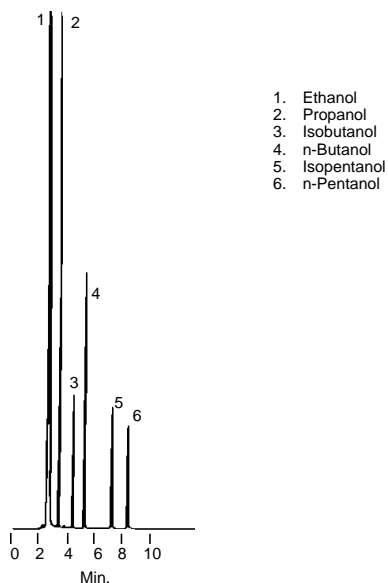
**Figure E. A Single Capillary Column Can Be Used in Analyses of Many Industrial Solvents**



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**Figure F. Symmetrical Peaks from Small Quantities of Alcohols, Using Capillary GC**

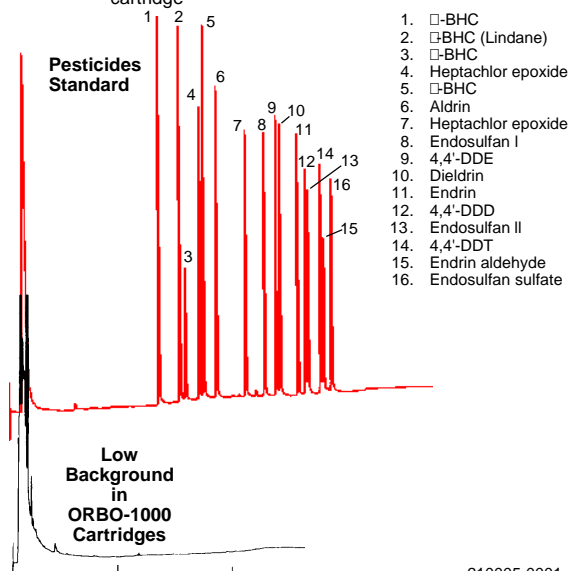
Column: **SPB-1701, 30m x 0.25mm, 0.25µm film**  
 Cat. No.: **24113**  
 Oven: **60°C**  
 Carrier: **helium, 20cm/sec (set at 60°C)**  
 Det.: **FID**  
 Inj.: **1µL alcohols blend, split (100:1) (2.5ng each compound on column)**



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**Figure H. Monitor Pesticides in Indoor Atmospheres**

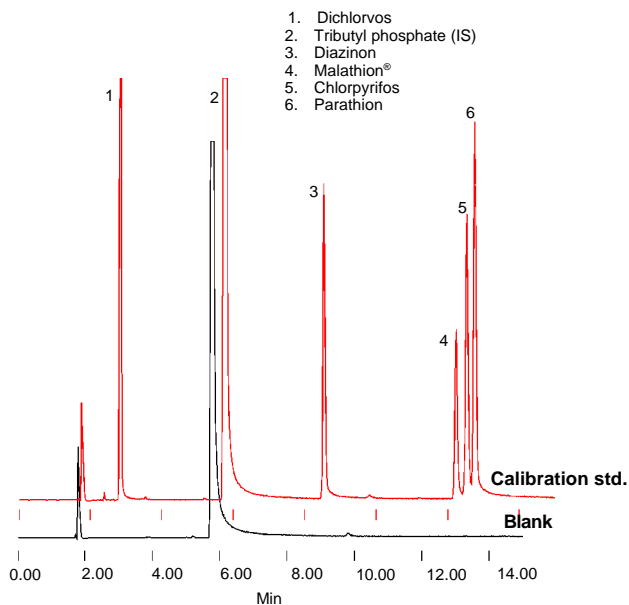
Column: **SPB-608, 30m x 0.53mm, 0.50µm film**  
 Cat. No.: **25312**  
 Oven: **150°C (1 min) to 280°C at 8°C/min, hold 12 min**  
 Carrier: **helium, 5mL/min**  
 Det.: **ECD**  
 Inj.: **4µL hexane (10ng/mL each analyte) or 4µL (15% ethyl ether in n-hexane) extract of unused PUF cartridge**



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**Figure G. Organophosphorous Pesticides by OSHA Method 62**

Sample: **calibration std.: 12µg/mL organophosphorous pesticides and 78µg/mL tributylphosphate (IS) in toluene**  
**blank: Supelpak 20 (XAD-2) and glass fiber filter desorbed in 78µg/mL tributylphosphate (IS) in toluene**  
 Sampling Tube: **ORBO-49P**  
 Cat. No.: **20350**  
 Column: **SPB-20, 60m x 0.53mm ID, 1.0µm film**  
 Cat. No.: **25390**  
 Oven: **200°C (2min) to 250°C at 4°C/min**  
 Carrier: **helium, 8mL/min**  
 Det.: **FPD, 300°C**  
 Inj.: **1µL, direct injection, 220°C (packed column injector)**



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**Figure I. Inorganic Acids Resolved at Low Levels, Using Suppressor Column Ion Chromatography and ORBO-53 Tube**

Column: **Dionex AS3 Fast Run Anion, 25cm x 3mm ID, with Fast Run precolumn**  
 Mobile Phase: **3mM NaHCO<sub>3</sub>/2.4mM Na<sub>2</sub>CO<sub>3</sub>**  
 Flow Rate: **3mL/min**  
 Det.: **conductivity (10µSiemens/cm)**  
 Inj.: **100µL**

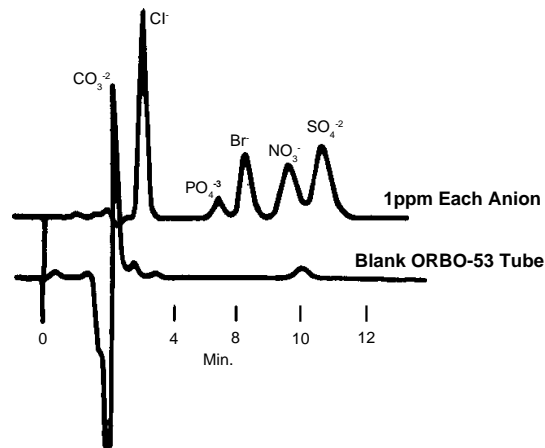


Figure provided by Ms. Mary Ellen Cassinelli, NIOSH Inorganic Methods Development Section, Cincinnati, OH USA.

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## Quantification

### Preparing Standards

An accurately calibrated chromatograph is critical to quantification of airborne contaminants. Calibration typically involves determining the linearity of detector response at multiple levels of solute concentration (e.g., 0.1, 0.5, 1, 2, and 5 times the OSHA exposure limit). To prepare such standards, use Equation 1 (vaporized liquid analytes) or Equation 2 (vaporized solid analytes) below. •

#### Equation 1:

Standard solution of a vaporized liquid analyte (expressed as  $\mu\text{L}$  in  $D$  mL of desorbing agent) =

$$(F) \frac{L \times V_s \times M}{24450 \times P \times D}$$

where

- F = fraction or multiple (e.g., 0.1, 0.5, 1, 2, or 5X) of OSHA-TWA limit
- L = OSHA-TWA limit, in ppm ( $\mu\text{L}$  of analyte/liters of air)
- $V_s$  = volume of air sampled, in liters (corrected to 25°C, 760mm Hg $\blacktriangle$ )
- M = molecular weight of analyte, in g/mole
- 24450 = molar volume, in mL/mole (at 25°C, 760mm Hg)
- P = density of analyte, in g/mL
- D = volume of desorbing agent, in mL

#### Equation 2:

Standard solution of a vaporized solid analyte (expressed as mg in  $D$  mL of desorbing agent) =

$$(F) \frac{L_c \times V_s}{D}$$

where

- F = fraction or multiple (e.g., 0.1, 0.5, 1, 2, or 5X) of OSHA-TWA limit
- $L_c$  = OSHA-TWA limit (mg of analyte/ $\text{m}^3$  of air $\blacksquare$ )
- $V_s$  = volume of air sampled, in  $\text{m}^3$  (corrected to 25°C, 760mm Hg $\blacktriangle$ )
- D = volume of desorbing agent, in mL

$\blacktriangle$ to correct temperature and pressure:

$$V_s = \text{volume air sampled} \times \frac{\text{pressure}}{760\text{mm}} \times \frac{298^\circ\text{K}}{\text{temp. in } ^\circ\text{C} + 273}$$

- The volume/volume and weight/volume concentrations can be easily interconverted:

$$\text{ppm to (x) mg/m}^3 = \frac{[(y) \text{ ppm}] (\text{mol. wt. of contaminant})}{24.5}$$

$$\text{mg/m}^3 \text{ to (y) ppm} = \frac{[(x) \text{ mg/m}^3] (24.5)}{\text{molecular weight of contaminant}}$$

- $1\text{m}^3 = 1000$  liters

- When working with highly volatile liquids such as chloroform and  $\text{CS}_2$ , inject the analyte and desorbing solvent into an ampul or a septum-sealed vial.

Use Equation 1 to convert the OSHA-TWA limit, L, (expressed as  $\mu\text{L}$  of vaporized liquid analyte/liter of sampled air) to expression as a liquid volume, encountered when the analyte is desorbed from the adsorbent into the desorbing agent. Similarly, Equation 2 converts  $L_c$  (expressed as mg of vaporized solid analyte per  $\text{m}^3$  or 1000 liters of sampled air) to expression as a weight/volume concentration, encountered when the analyte is desorbed from the adsorbent into the desorbing agent.

The following examples illustrate the preparation of standards for analyses of chloroform (liquid) and chlordane (solid) vapors. •

**Example 1:** Prepare a standard solution of chloroform in 1mL of  $\text{CS}_2$  at 0.5X the OSHA-TWA limit. The limit is 50ppm, or 50 $\mu\text{L}$  chloroform/liter of air.

Fit the physical properties of chloroform and the air sample volume (from the appendix) into Equation 1:

$$\frac{\square \text{L CHCl}_3}{\square \text{L CS}_2} = (0.5) \frac{\left(\frac{50 \square \text{L CHCl}_3}{\text{liters air}}\right) \left(\frac{10}{\text{liters air}}\right) \left(\frac{119.4\text{g}}{\text{mole}}\right)}{\left(\frac{24450 \text{ mL}}{\text{mole}}\right) \left(\frac{1.48\text{g}}{\text{mL}}\right) \left(1\text{mL CS}_2\right)}$$

Transpose the denominator to the numerator by inverting, and cancel terms:

$$\frac{\mu\text{L CHCl}_3}{\text{mL CS}_2} =$$

$$(0.5) \left(\frac{50\mu\text{L}}{\text{liters air}}\right) \left(\frac{10}{\text{liters air}}\right) \left(\frac{119.4\text{g}}{\text{mole}}\right) \left(\frac{1\text{mL}}{1.48\text{g}}\right) \left(\frac{1\text{mole}}{24450\text{mL}}\right) \left(\frac{1}{1\text{mL CS}_2}\right) = \frac{250 \times 119.4\mu\text{L CHCl}_3}{14.8 \times 24450\text{mL CS}_2} = \frac{0.82\mu\text{L CHCl}_3}{1\text{mL CS}_2}$$

Therefore, you need 0.82 $\mu\text{L}$  of chloroform in 1mL of  $\text{CS}_2$  to prepare a 25ppm chloroform standard. Substituting 1, 2, and 5 for F in Equation 1 will give you a total of four standard solutions to establish a calibration curve. Alternatively, to minimize sampling errors, prepare the 2, 1, and 0.5X standards from the 5X standard by dilution. To minimize error due to the volatility of these liquids, prepare 10mL of each standard, rather than 1mL.

**Example 2:** Prepare a standard solution of chlordane in 10.0mL of toluene at 5X the OSHA-TWA limit. The limit is 0.5mg chlordane/ $\text{m}^3$  of air.

Fit the appropriate values (obtain the sample volume from the appendix) into Equation 2:

$$\frac{\text{mg chlordane}}{\text{mL toluene}} = (5) \frac{0.5\text{mg/m}^3 \times (120\text{L} \times 1 \text{m}^3/1000\text{L})}{10\text{mL}} = \frac{0.3\text{mg}}{10\text{mL}}$$

Therefore, 0.3mg of chlordane in 10mL of toluene represents 2.5mg of chlordane in  $1\text{m}^3$  of air, a standard of 5X the OSHA-TWA limit. Substituting 2, 1, and 0.5 for F in Equation 2 will give you a total of four standard solutions to establish a calibration curve for chlordane. Alternatively, to minimize weighing errors, prepare the 2, 1, and 0.5X standards from the 5X standard by dilution.

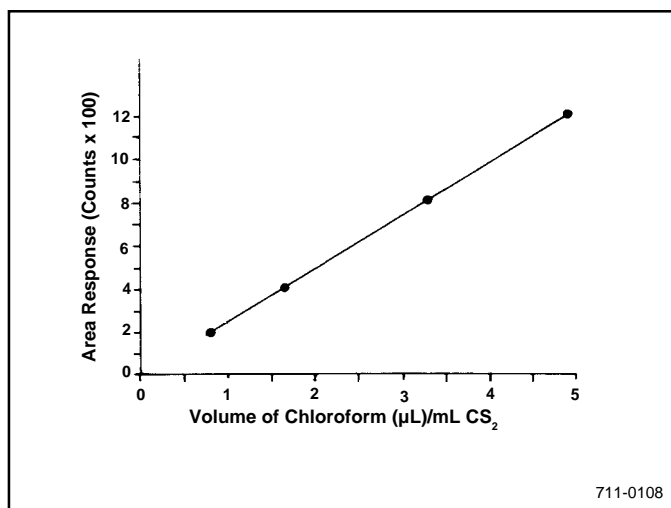
### Calibration Curves

Aliquots of the standards must be injected in the chromatograph under the same run conditions used in the sample analysis. Calibration curves are prepared by plotting peak area of the analyte versus analyte concentration. Figure J is an example of a calibration curve for chloroform, prepared using the chloroform volumes calculated from Equation 1. Figure K is an example of a calibration curve for chlordane, prepared using the chlordane amounts calculated from Equation 2.

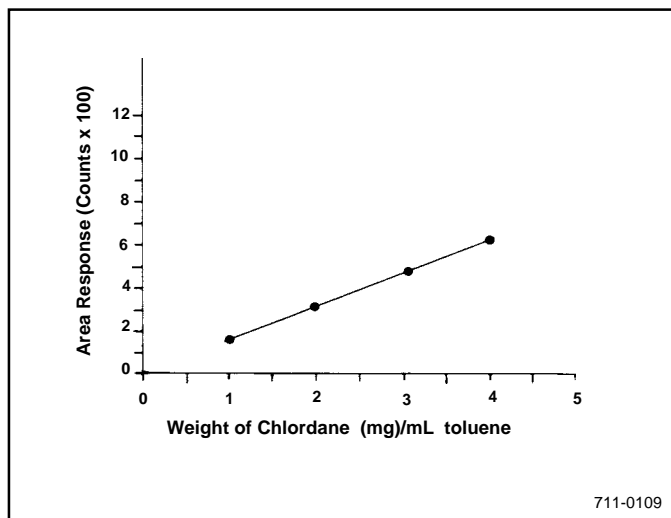
### Sample Analysis

To determine the concentration of the analyte in the desorbing solution, compare the analyte peak area from the chromatogram to the calibration curves. The concentration in the air sample then can be calculated. The following examples for chloroform and chlordane illustrate the necessary steps. Area responses for blanks must also be converted to airborne analyte concentrations and subtracted from the sample concentrations. These blanks must measure the background of both the adsorbent and the desorbing solution.

**Figure J. Calibration Curve for Chloroform, Using Standards Prepared from Equation 1**



**Figure K. Calibration Curve for Chlordane, Using Standards Prepared from Equation 2**



Example (chloroform): The area response (counts) for chloroform is directly related to the concentration of solvent ( $\mu\text{L}$  chloroform/ $\text{mL}$   $\text{CS}_2$ ). Convert solvent/volume of air to ppm (atmospheric concentration), using Equation 3:

### Equation 3:

$$\text{ppm} = \frac{\mu\text{L}}{\text{liter}} = \frac{\mu\text{L of liquid analyte} \times 24450 \times P \times D}{V_s \times M}$$

where

- 24450 = molar volume, in mL/mole (at 25°C, 760mm Hg\*)
- P = density of contaminant, in g/mL
- D = volume of desorbing agent, in mL
- $V_s$  = volume of air sampled, in liters (corrected to 25°C, 760mm Hg\*)
- M = molecular weight of analyte, in g/mole

To convert 1.0 $\mu\text{L}$  of chloroform in a sample solution to ppm of chloroform in the air sample, assuming a 10-liter air sample and 1 mL  $\text{CS}_2$  used in desorption:

$$\begin{aligned} \frac{\text{ppm} (\mu\text{L})}{\text{liter}} &= \frac{\left(\frac{1.0\mu\text{L CHCl}_3}{1\text{mL CS}_2}\right) \left(\frac{24450\text{mL}}{\text{mole}}\right) \left(\frac{1.48\text{g}}{\text{mL}}\right) (1\text{mL CS}_2)}{\left(\frac{10}{\text{liters air}}\right) \left(\frac{119.4\text{g}}{\text{mole}}\right)} \\ &= \left(\frac{1.0\mu\text{L CHCl}_3}{1\text{mL CS}_2}\right) \left(\frac{24450\text{mL}}{\text{mole}}\right) \left(\frac{1.48\text{g}}{\text{mL}}\right) (1\text{mL CS}_2) \left(\frac{1}{10}\right) \left(\frac{\text{mole}}{119.4\text{g}}\right) \\ &= \frac{1.0 \times 24450 \times 1.48 \text{ g} \times \mu\text{L CHCl}_3}{10 \times 119.4 \text{ liters air}} = \frac{30.5\mu\text{L CHCl}_3}{\text{liters of air}} \\ &= 30.5\text{ppm CHCl}_3 \text{ in air} \end{aligned}$$

Example (chlordane): The area response (counts) for chlordane is directly related to the concentration of solid (mg chlordane/ $\text{mL}$  toluene). Convert solid/volume of air to  $\text{mg}/\text{m}^3$  (atmospheric concentration) by using Equation 4:

### Equation 4:

$$\text{mg}/\text{m}^3 = \frac{\text{mg}/\text{mL solid in desorbing solvent}}{V_s} \times D$$

where

- D = volume of desorbing solvent, in mL
- $V_s$  = volume of air sampled, in  $\text{m}^3$  or liters<sup>▲</sup> (corrected to 25°C, 760mm Hg\*)

To convert 0.1mg of chlordane in 1mL solution to  $\text{mg}/\text{m}^3$  of chlordane in the air sample, assuming a 120 liter air sample and 10mL of toluene used in desorption:

$$\text{mg}/\text{m}^3 = \frac{0.1\text{mg}/\text{mL}}{120\text{L} \times 1\text{m}^3/1000\text{L}} \times 10\text{mL} = 8.33\text{mg}/\text{m}^3 \text{ chlordane in air}$$

▲1 $\text{m}^3$  = 1000 liters

\*See Equation 1

Each airborne analyte investigated, whether gaseous or particulate, is quantified in the same manner, using the formulas stated above and the appropriate calibration curve.

Because an absolute calibration is used, standard and sample analyses must be performed under the same conditions. Precautions must be taken to prevent error-causing changes in samples and standards stored for more than a day:

volatilization of the analyte from the desorbing solvent, which produces a false (low) value for the analyte's concentration in the air sample

volatilization of the desorbing solvent, which produces a false (high) value for the analyte's concentration in the air sample

sample contamination (via loose vial cap, unclean cap liner, or other means)

#### *Evaluating Collection and Desorption Efficiency*

When monitoring airborne contaminants, consider and correct for the collection efficiency of the adsorbent or filter and the desorption efficiency of the desorbing solvent for the specific analyte. The appendix lists the recommended collection device and flow rate for sampling each airborne contaminant. Where the data are available, TWA, STEL, and ceiling values are listed.

Prepare desorption efficiency standards from unexposed sample collection devices. Place the adsorbent beds or filters in septum cap vials, then inject through the septum cap the quantity of analyte representing the OSHA-TWA limit in the specified air sample volume (use the appendix and Equations 1 and 2; dissolve solid analyte in a small amount of a suitable solvent). Also evaluate several other concentrations of analyte, to determine if desorption efficiency varies with analyte concentration (4). To establish a value for 100% desorption, inject the OSHA-TWA limit of the analyte into empty vials (vial blanks). Evaluate additional sampling devices for background contamination or permeation of the septum (adsorption blanks).

Allow the sealed vials to stand overnight. This will enable the analyte and adsorbent to achieve equilibrium. Desorb and analyze the efficiency standard samples, vial blanks, and adsorption blanks in exactly the same manner, as previously described in this bulletin. Determine desorption efficiency as follows:

$$\text{Desorption Efficiency (\%)} = \frac{\text{area}_{\text{(efficiency standard sample)}} - \text{area}_{\text{(adsorption blank)}}}{\text{area}_{\text{(vial blank)}}} \times 100$$

To correct for less than 100% desorption, divide the value you obtain for the analyte in on-site air samples by the desorption efficiency value (e.g., 99%).

#### **Calibrating the Personal Pump and Determining Pressure Drop**

To ensure accurate air sampling, the personal air sampling pump must be calibrated daily, prior to sampling. Insert the appropriate sampling device(s) in the sampling position, and set the pump flow. Measure the flow with a flow-bubble meter upstream of the sampling device. If necessary, adjust the flow. It is also good practice to recheck the flow *after* sampling.

The pressure drop across the sampling device should also be monitored. A large pressure drop will excessively strain the pump and could cause the batteries to wear down before the end of the sampling period. Determine pressure drop by connecting a mercury or water manometer in series with the pump and sampling device. Maximum pressure drops, where applicable, are listed in the individual NIOSH and OSHA methods. Because ORBO adsorbent tubes are tested to ensure the pressure drop complies with NIOSH and OSHA recommendations, you need not sacrifice time or tubes in assessing this parameter.

#### **Limitations of the Adsorbent Tube and Filter Sampling Methods**

Adsorbent tubes and filters are extremely reliable for collecting airborne contaminants, but please note the following limitations:

Air sampling devices have unique capacities for each analyte sampled. When this capacity is exceeded, analyte breakthrough occurs. Air sampling recommendations must be followed.

An analyte of interest can be displaced by another analyte more strongly adsorbed by the adsorbent.

High humidity can severely reduce the breakthrough volumes of adsorbents like charcoal and silica gel. Other adsorbents, such as Carboxen and Supelpak 20 adsorbents, usually are unaffected by moisture.

The desorbing solvent recommended in a specific procedure may not always displace the analyte from the adsorbent. If necessary, choose a more effective solvent.

Reactive analytes can be converted to other species during the collection process, hence quantitation may be inaccurate.

To ensure best results from the sampling method, refer to the appendix for the correct adsorbent device, sampling procedure, and analytical technique.

#### **References**

1. *NIOSH Manual of Analytical Methods*, 4th Edition, August 1994, DHHS Publication No (NIOSH) 94-113.
  2. Occupational Safety and Health Administration (OSHA) standards (Title 29, *Code of Federal Regulations*, Part 1910, Subpart Z, Toxic and Hazardous Substances, Section 1910.1000, Air Contaminants, revised July 1, 1985.)
  3. Jenke, D.R., P.K. Mitchell and G.K. Pagenkopf, *J. Chromatogr. Sci.*, 21, 487 (1983).
  4. Hill, R.H., Jr., C.S. McCammon, A.T. Saalwaechter, A.W. Teass and W.J. Woodfin, *Anal. Chem.*, 48, 1395 (1976).
- Reference 1 available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

## Ordering Information:

### ORBO Solvent Desorption Sampling Devices

ORBO No.	Adsorbent	Bed Wt. (mg) A/B	Dimensions (mm) OD x Length	SKC Inc. Equivalent	Qty.	Cat. No.
<b>Charcoal</b>						
32 large	Activated coconut charcoal (20/40)	400/200	8 x 100	226-09	50	20228
32 small	Activated coconut charcoal (20/40)	100/50	6 x 70	226-01	50	20267
33	Activated petroleum charcoal (20/40)	700/390	8 x 150	226-36	50	20259
34	Activated coconut charcoal, specially treated	400/200	8 x 100	—	25	20211
301	Charcoal (20/40)	150	6 x 70	—	50	20039 ■
303	Charcoal (petroleum) (20/40)	100/50	6 x 70	226-38	50	20040 ■
304	Charcoal (low Ni) (20/40)	120/60	6 x 80	—	50	20041 ■
306	Charcoal (petroleum) (20/40)	400/200	8 x 110	—	50	20073
351	4-tert-butyl catechol on charcoal	100/50	6 x 70	226-73	50	20042 ■
352	Oxidizer-impregnated charcoal	100/50	6 x 70	—	50	20043 ■
353	HBr on petroleum charcoal	100/50	6 x 70	226-38-03	25	20044 ■
354	Alkali-treated charcoal (AVL Barneby Cheney, 580-19)	100/50	6 x 70	226-67	50	20045 ■
355	4-tert-butyl catechol on charcoal	110/55	6 x 70	—	50	20046 ■
356	4-tert-butyl catechol on charcoal	400/200	8 x 110	—	50	20047 ■
<b>Carbon</b>						
91	Carbosieve S-III carbon molecular sieve <sup>♦</sup>	130/65	6 x 70	226-121	25	20360
90	Carboxen-564 <sup>▼</sup> carbon molecular sieve <sup>*</sup>	160/80	6 x 70	226-81	25	20358
92	Carboxen-564 carbon molecular sieve <sup>*</sup>	160/80	6 x 70	226-81	25	20362
78	HBr on Carboxen-564 carbon molecular sieve	400/200	6 x 110	—	25	20355
100	Carbotrap (20/40)	350/175	7 x 110	—	25	20255
101	Carbotrap (20/40)	100/50	6 x 70	—	25	20254
1004	KOH-coated carbon	1.5g	9 x 110	226-31	20	20035 ■
77	H <sub>2</sub> SO <sub>4</sub> -treated carbon bead (20/30)	500/250	8 x 110	226-29	50	20036 ■
<b>Silica Gel</b>						
52 small	Activated silica gel (20/40)	150/75	6 x 70	226-10	50	20229
52 large	Activated silica gel (42/60)	150/150	8 x 70	226-48	50	20263
507	Silica gel (20/40)	520/260	8 x 110	226-15	50	20870
53	Activated silica gel (20/40) with glass fiber filter	400/200	7 x 100	226-10-3	50	20265
LpDNPH S10	2,4-DNPH on silica (SPE cartridge)	350	11 x 74	—	10	21024
502	Activated silica gel (20/45)	100/50	6 x 70	—	50	20030 ■
504	Activated silica gel (45/60)	150/75	6 x 70	—	50	20031 ■
506	Activated silica gel (45/60)	300/150	8 x 70	226-10-04	50	20032 ■
554	H <sub>2</sub> SO <sub>4</sub> -coated silica gel (20/40)	150/75	6 x 70	226-53	50	20033 ■
<b>Florisil<sup>®</sup></b>						
60	Florisil (30/45)	100/50	6 x 70	—	50	20351
<b>Hopcalite</b>						
1002	Hopcalite	200	6 x 70	226-17-1A	50	20863
1008	Hopcalite	500	8 x 110	226-17-3A	50	20866
<b>Porous Polymers</b>						
42 small	Supelpak 20E (20/40)	66/33	6 x 70	226-49-102	50	20262
42 large	Supelpak 20P (20/40) <sup>*</sup>	100/50	6 x 70	226-30-04	50	20264
43	Supelpak 20U (20/40) <sup>*</sup>	100/50	6 x 70	226-30-04	50	20258
44	Supelpak 20E (20/40) <sup>*</sup>	100/50	6 x 70	226-30-04	50	20260
	with glass fiber filter (OVS-2)	270/140	—	226-30-16	10	20350
23	2-(Hydroxymethyl)piperidine on Supelpak 20N (20/40)	120/60	6 x 70	226-118	25	20257
	on Supelpak 20N (20/40)	150/75	6 x 100	226-117	25	20231

■ These tubes are made to order.

<sup>♦</sup>German Pat. No. 1935500. Patent holder — Badische Anilin- & Soda-Fabrik Aktiengesellschaft.

<sup>▼</sup>US Pat. No. 4,839,331.

<sup>\*</sup> Similar tubes QA tested for different methods. Refer to *Appendix to Technical Bulletin 769* (request from customer service).

### Custom Tubes

In addition to our stock product and made-to-order offering, we have the capability to make many tubes to your exact specifications. Perhaps you are outgrowing your internal tube-making capability, or you need a tube listed in an OSHA or NIOSH method that is not a stock product. Maybe you have a special project for which you need tubes immediately. Whatever your reason, we can meet your custom tube needs. See page 10 for ordering information.



## ORBO Solvent Desorption Sampling Devices

ORBO No.	Adsorbent	Bed Wt. (mg) A/B	Dimensions (mm) OD x Length	SKC Inc. Equivalent	Qty.	Cat. No.
<b>Porous Polymers (contd. )</b>						
25	2-(Hydroxymethyl)piperidine on Supelpak 20N (20/40)	450/225	8 x 100	226-27	25	20357
70	5% Na <sub>2</sub> CO <sub>3</sub> on Chromosorb P (20/40)	335/185	8 x 100	—	50	20256
47	Supelpak 70	100/50	6 x 70	226-95	50	20349
402	Tenax TA 35/60	100/50	8 x 110	226-35-03	50	20832
403	Tenax TA 60/80, acetone/methanol-treated	100/50	6 x 70	—	50	20034 ■
601	Amberlite XAD-8 (16/50)	100	6 x 70	226-30-08	50	20048 ■
605	Amberlite XAD-2 (20/50)	100/50	6 x 70	226-30-04	50	20049 ■
608	Amberlite XAD-2 (20/50)	150/75	8 x 110	226-30-05	50	20050 ■
609	Amberlite XAD-2 (20/50)	400/200	8 x 110	226-30-06	50	20051 ■
613	Amberlite XAD-4	80/40	6 x 70	226-93	50	20052 ■
615	Amberlite XAD-7 (15/50)	100/50	6 x 70	226-95	50	20053 ■
655	Amberlite XAD-7 (20/60), H <sub>3</sub> PO <sub>4</sub> (phosphoric acid)-treated	80/40	6 x 70	226-98	—	20054 ■
657	Amberlite XAD-7, 1-(2-pyridyl)piperazine-coated	80/40	6 x 70	—	50	20055 ■
706	Chromosorb 102 (20/40)	100/50	8 x 100	226-107	50	20057 ■
710	Chromosorb 108 (60/80)	400/200	10 x 100	226-113	50	20058 ■
711	Chromosorb 106 (60/80)	600/300	10 x 100	—	50	20059 ■
751	Silvered Chromosorb P	30	6 x 20	226-17-4	50	20060 ■
1101	Porapak QS (50/80)	100/50	6 x 70	226-59-09	50	20061 ■
1102	Porapak P (50/80)	100/50	6 x 70	226-114	50	20062 ■
1103	Porapak Q (50/80)	150/75	6 x 100	226-115	50	20063 ■
1104	Porapak R (50/80)	70/35	6 x 70	226-59-04	50	20064 ■
<b>Polyurethane Foam</b>						
1000	Polyurethane foam (PUF) cartridge	—	22mm x 7.6cm <sup>▲</sup>	226-92	3	20557
—	Glass holder for PUF	—	—	—	1	20556
—	Precleaned PUF plug	—	—	—	3	20600
—	PUF filter cartridge assembly (replacement parts available)	—	22mm x 7.6cm <sup>▲</sup>	—	1	21031
2000	Large PUF cartridge	—	7.0006cm x 6cm <sup>▲</sup>	226-131	1	20037 ■
—	Glass holder for Large PUF	—	—	—	1	20563
—	Precleaned Large PUF plug	—	—	—	1	20038
<b>Coated Filters</b>						
80	1-(2-Pyridyl)piperazine on glass fiber filter	—	37mm	—	25	20811
80 kit	ORBO-80 filter with cassettes, unassembled	—	37mm	225-9002	25	20812
822	H <sub>2</sub> SO <sub>4</sub> -coated glass fiber filter	—	37mm	—	25	20065 ■
823	KOH/glycerine-coated glass fiber filter	—	37mm	—	25	20066 ■
824	Na <sub>2</sub> CO <sub>3</sub> impregnated GN-4 mixed cellulose ester membrane	—	37mm	—	25	20067 ■
826	Mercuric acetate-coated A/E glass fiber	—	37mm	—	25	20068 ■
827	DNPH and phosphoric acid-coated A/E glass fiber	—	37mm	—	25	20069 ■
828	Silver nitrate - impregnated Whatman #4	—	37mm	—	25	20070 ■
829	0.1mg 1-(2-Pyridyl) piperazine on glass fiber filter	—	37mm	—	25	20071 ■
834	2 mg 3,4-Dimethoxybenylamine on A/E glass fiber	—	37mm	—	25	20072 ■

■ These tubes are made to order.

▲ Dimensions are for the PUF plug.

**Please Note:** We cannot ensure the purity of adsorbents we do not manufacture or do not normally stock. Made-to-order adsorbent tubes are not routinely tested, but testing can be done on request.

# Custom Air Monitoring Tubes

How To Order

## Custom Air Monitoring Tube Order Form

To order: Fax or mail us a copy of this completed form, or phone with the requested information. Phone **800-247-6628** or **814-359-3441**, or FAX **800-447-3044** or **814-359-3044**.

Name: \_\_\_\_\_

Address: \_\_\_\_\_

Phone/Fax: \_\_\_\_\_

Solvent Desorption

Thermal Desorption

Repeat Order (Previous MR# \_\_\_\_\_ )

Quote Number: \_\_\_\_\_

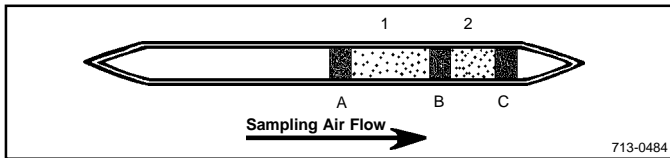
Method Number : \_\_\_\_\_

Analyte: \_\_\_\_\_

No. of Tubes Ordered: \_\_\_\_\_

### Tube Specifications

#### Solvent Desorption



- 6mm OD glass
- 7mm OD glass
- 8mm OD glass
- 10mm OD glass
- Other \_\_\_\_\_
- Length \_\_\_\_\_

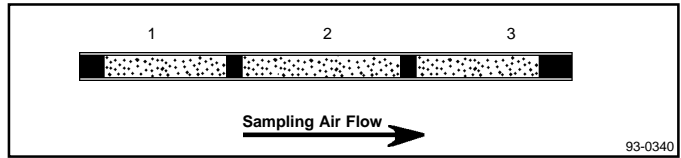
#### Retaining Plugs:

- |   |                            |                            |                            |
|---|----------------------------|----------------------------|----------------------------|
| Silanized glass wool                          | <input type="checkbox"/> A | <input type="checkbox"/> B | <input type="checkbox"/> C |
| Glass wool                                    | <input type="checkbox"/> A | <input type="checkbox"/> B | <input type="checkbox"/> C |
| Foam  | <input type="checkbox"/> A | <input type="checkbox"/> B | <input type="checkbox"/> C |
| <input type="checkbox"/> Stainless steel clip |                            |                            |                            |

#### Adsorbent:

1. \_\_\_\_\_
2. \_\_\_\_\_
3. \_\_\_\_\_
4. \_\_\_\_\_

#### Thermal Desorption<sup>▲</sup>



- Glass
- Stainless Steel

#### Dimensions:

OD \_\_\_\_\_ ID \_\_\_\_\_ Length \_\_\_\_\_

Length heated zone on instrument \_\_\_\_\_

#### Retaining plugs:

- Fritted (in glass)
- Glass wool
- Screens (in stainless steel)
- Stainless Steel Clip (in glass)

TDU Instrument: Model No. \_\_\_\_\_

- C D S Analytical
- Chrompak
- Dynatherm
- Envirochem
- O.I. Analytical
- Perkin Elmer
- Tekmar

#### Bed weights :

1. \_\_\_\_\_
2. \_\_\_\_\_
3. \_\_\_\_\_
4. \_\_\_\_\_

#### Comments:

\_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

<sup>▲</sup> One, two, three, and four beds available

## Air Monitoring Products from Supelco



996-0283

Supelco's air sampling products are designed for use in industrial hygiene, indoor and ambient air monitoring, and source emission testing. We manufacture our specialty adsorbent tubes in our own facilities, using the latest technology in sorbent purity. All adsorption tubes are designed and prepared to conform to NIOSH, OSHA, EPA, and ASTM specifications for adsorbent quality, divider composition and pore size, tube dimensions, and back pressure. We scrupulously clean the glass tubing, purify all adsorbents and other components, and prepare the tubes in a contaminant-free environment. All raw materials, as well as every lot of finished tubes, must pass stringent QA testing.

The proper sample collection device is crucial to obtaining reliable data for regulatory compliance. The ideal sampling device must trap the analyte (air pollutant) of interest in all of the various physical states in which it can exist: vapor, aerosol, and particulate. In addition, the ideal device must ensure minimal background interference and sufficient capacity or breakthrough volume for the application in which it will be used.

In general, solvent desorption tubes are used for industrial hygiene applications. They are desorbed or extracted with an organic solvent and an aliquot of the extract is analyzed by GC or HPLC. Thermal desorption tubes are used primarily for sampling ambient and indoor air. A thermal desorption unit (TDU) is used to rapidly heat the tube to desorb the analytes of interest and transfer them directly to a gas chromatograph. Unlike solvent desorption tubes, thermal desorption tubes can contain beds of different adsorbents, allowing the collection of a wide range of compounds. Because *all* of the trapped analyte is desorbed and analyzed (not just an aliquot, as in solvent desorption), sensitivity can be enhanced up to a thousandfold over solvent desorption analyses. Thermal desorption tubes also can be reconditioned and reused.



994-0232

Refer to the current Supelco catalog for these sample collection devices and equipment:

- Solvent Desorption Tubes (ORBO Tubes)
  - activated charcoal
  - carbon: Carbotrap™, Carboxen™, Carbosieve™
  - silica gel
  - Florisil®
  - porous polymers: Supelpak™
  - polyurethane foam
- Thermal Desorption Tubes
  - carbon molecular sieve: Carboxen, Carbosieve
  - graphitized carbon: Carbopack, Carbotrap
  - porous polymer: Tenax®, Chromosorb®
- Filters — for particles and aerosols
- Gas Sampling Bags — for whole air samples
- Passive Monitors — no sampling pump needed
- Impingers — liquid sampling media
- Personal Sampling Pumps
- 24-Hour Sequential Sampler
- Vacuum Box Bag Samplers
- Ambient Air Samplers

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