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GAS AND VAPOR SAMPLE COLLECTORS

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Introduction

This chapter discusses the collection and analysis of gases and vapors commonly found in the industrial or workplace environment. It is limited to descriptions of sampling methods for subsequent laboratory analysis. It does not, therefore, include any discussions of direct-reading instruments, colorimetric indicators, tape samplers, and other “on-the-spot” testing devices.

Nature of Industrial Gases and Vapors

The terms gases and vapors are frequently used interchangeably; however, they are not identical. The majority of gases of interest to the industrial hygienist are elements, e.g., chlorine, or inorganic compounds, e.g., hydrogen cyanide, ammonia, arsine, and carbon monoxide. Vapors of industrial importance are mainly organic substances such as methyl ethyl ketone, benzene,

acetone, toluene, and toluene diisocyanate, although some inorganic substances, e.g., mercury, are also encountered.

While it is true that, at ordinary temperature and pressure, gases and vapors will both diffuse rapidly and form true solutions in air, they differ in other respects. Gases are generally understood to be noncondensable at room temperature and vapors to be derived from volatile liquids. Therefore, under ordinary conditions, gases exist in the gaseous state even when present at high concentrations. Vapors, on the other hand, may condense at high concentrations and coexist in both gas and aerosol forms. However, unless an aerosol is deliberately produced as in a spray operation, atmospheric concentrations of vapor pollutants rarely reach saturation conditions; gases and vapors can then be considered similar and the same devices used to collect them.

Sampling Procedures

There are two basic methods for collecting gaseous samples. In one, called grab sampling, an actual sample of air is taken in a flask, bottle, bag, or other suitable container; in the other, called continuous or integrated sampling, gases or vapors are removed from the air and concentrated by passage through an absorbing or adsorbing medium.

The first method involves the collection of grab or instantaneous samples, usually within a few seconds or a minute. This type of sampling is acceptable when peak concentrations are sought or when concentrations are relatively constant. Grab samples were once used only for gross components of gases, such as methane, carbon monoxide, or oxygen, where the analysis was frequently performed volumetrically. The introduction of highly sensitive laboratory instruments, however, makes this technique limited only by the detection limit of the analytical methods available. Vinyl chloride, for example, is measurable in grab samples by gas chromatography at levels well below 1.0 ppm.

An important feature of grab samples is that their collection efficiency is normally 100%. However, it must be remembered that sample decay does occur for various reasons, and grab sampling must be used with this clearly in mind.

Grab sampling is of questionable value when 1) the contaminant or contaminant concentration varies with time, 2) the concentration of atmospheric contaminants is low, or 3) a time-weighted average exposure is desired. In such circumstances, continuous or integrated sampling is used instead. The gas or vapor in these cases is extracted from air and concentrated by 1) solution in an absorbing liquid, 2) reaction with an absorbing solution (or reagent therein), or 3) collection onto a solid adsorbent. Collection efficiency of active

sampling devices utilized for these sampling procedures is frequently less than 100%; therefore, individual efficiency percentages must be determined for each case. Later in this chapter (and in *Chapters R* and *T*), another technique, passive (or diffusive) sampling, is discussed.

Selection of Sampling Devices

The first step in the selection of a sampling device and analytical procedure is to search the available literature. Primary sources are the compendia of methods recommended by the regulatory authorities, i.e., the *NIOSH Manual of Analytical Methods*⁽¹⁾ and the *OSHA Analytical Methods Manual*.⁽²⁾ Recommended methods from other countries, such as the United Kingdom,⁽³⁾ Germany,⁽⁴⁾ or Sweden,⁽⁵⁾ might also be consulted. Secondary sources are published literature references in, for example, the *American Industrial Hygiene Association Journal*, *Applied Industrial Hygiene*, or *Analytical Chemistry*, or books such as the Intersociety Committee's *Methods for Air Sampling and Analysis*.⁽⁶⁾

If a published procedure is not available, one can be devised from theoretical considerations. However, its suitability must be established experimentally before application. Important criteria for selecting sampling devices are solubility, volatility, and reactivity of the contaminant and the sensitivity of the analytical method.

Generally speaking, nonreactive and nonabsorbing gaseous substances may be collected as grab samples. Water soluble gases and vapors and those that react rapidly with absorbing solutions can be collected in simple gas washing bottles. Volatile and less soluble gaseous substances and those that react slowly with absorbing solutions require more liquid contact. For these substances, more elaborate sampling devices may be required such as gas washing bottles of the spiral type or fritted bubblers. Insoluble and nonreactive gases and vapors are collected by adsorption onto activated charcoal, silica gel, or other suitable adsorbent. Frequently, for a given contaminant, there may be several choices of sampling equipment.

Grab Samplers

Evacuated Flasks

These are heavy-walled containers of varying capacity and configurations. In each case, the internal pressure of the container is reduced. These containers are generally removed to a laboratory for analysis although it is possible to achieve field readability if the proper equipment and direct-reading instrument are available. Some examples of evacuated flasks are heavy-walled containers, separation flasks, and various commercial devices. These are described at the end of this chapter.

Gas or Liquid Displacement Containers

Any ordinary, sealable container can be used as a displacement sampler. Original air is replaced by test air by pumping or aspirating through the container with a double-acting rubber bulb aspirator or a battery- or electrically-operated vacuum pump. The volume of air swept out should be 10 to 15 times the container volume to achieve a sample collection efficiency of more than 99%. This is mathematically expressed by:⁽⁷⁾

$$N = 2.303 \log \left(\frac{100}{E} \right) \quad (1)$$

where: N = number of bottle volumes swept out
E = percent error in sample collection efficiency

An alternative method for sampling with these containers is to fill them with water and allow the water to drain out slowly in the test area. The liquid becomes replaced by test air. Obviously, this procedure is not suitable for collecting water-soluble gases.

For soluble and reactive gases, an absorbent or reagent solution may be introduced into the gas displacement sampler. The usual procedure is to fill the sampler with test air and then add the absorbent. When dealing with partially or totally evacuated flasks, the reagent solution or absorbent is added before they are put under pressure. In both cases, after the sample has been taken, the container is rotated to ensure an even distribution of the reagent on the inside surface of the sampler. This may take a few minutes or overnight, and so the equilibration time must be determined experimentally.

Flexible Plastic Containers

Plastic bags are used to collect air samples and prepare known concentrations which can range from parts per billion to more than 10% by volume in air. The bags are commercially available in sizes up to 250 L. However, 5- to 15-L bags are the most useful to industrial hygienists.

These bags are constructed from a number of plastic materials including polyester, polyvinylidene chloride, Teflon®, or other fluorocarbons. Plastic bags have the advantages of being light, nonbreakable, inexpensive to ship, and simple to use. But they should be used with caution since storage stabilities for gases, memory effects from previous samples, permeability, precision, and accuracy of sampling systems vary considerably.

Plastic bags should be tested before they are used. Some general recommendations are available in the published literature for the use of such bags for air sampling.^(6,8-10) A good review of specific applications up to 1967 is Schuette;⁽¹¹⁾ other specific applications are listed in Table S-1. Posner and Woodfin⁽¹⁸⁾ made a useful systematic study of five bag types and six organic vapors; they conclude that Tedlar® bags are best for

TABLE S-1. Some Storage Properties of Gases and Vapors in Plastic Bags

| Gas or Vapor | Bag Type | Reference |
|--|---|-----------|
| Various | Various | 11 |
| Vinyl chloride | Aluminized | 12 |
| Hydrocarbons | PVF | 13 |
| Vinyl chloride | Tedlar | 14 |
| Hydrocarbons | Saranex, Wine, Mylar Tedlar | 15 |
| Benzene | Tedlar | 16 |
| Chlorinated hydrocarbons | Tedlar | 17 |
| Methanol, acetone, benzene, butadiene, butene, trichloroethylene | Saran, Teflon, Halar, Tedlar, Aluminized | 18 |

short-term sampling, while aluminized bags are better for long-term storage prior to analysis. Storage properties, decay curves, and other factors, however, will vary considerably from those reported for a given gas or vapor since sampling conditions are rarely identical. Each bag, therefore, should be evaluated for the specific gas or gas mixture for which it will be used.

Continuous Active Samplers

Absorbers

The absorption theory of gases and vapors from air by solution, as developed by Elkins *et al.*,⁽¹⁹⁾ assumes that gases and vapors behave like perfect gases and dissolve to give a perfect solution. The concentration of the vapor in solution is increased during air sampling until an equilibrium is established with the concentration of vapor in the air. Absorption is never complete, however, since the vapor pressure of the material is not reduced to zero but is only lowered by the solvent effect of the absorbing liquid. Some vapor will escape with continued sampling, but it is replaced. Continued sampling, however, will not increase the concentration of vapor in solution once equilibrium is established.

According to formulae developed by Elkins *et al.*,⁽¹⁹⁾ introduced again by Neale and Perry and verified by Gage⁽²⁰⁾ in his experiments with ethylene oxide, the efficiency of vapor collection depends on: 1) the volume of air sampled, 2) the volume of the absorbing liquid, and 3) the volatility of the contaminant being collected. Efficiency of collection, therefore, can be increased by cooling the sampling solution (reducing the volatility of the contaminant), increasing the solution volume by adding two or more bubblers in series, or altering the design of the sampling device. Sampling rate and concentration of the vapor in air are not factors that determine collection efficiency.

Absorption of gases and vapors by chemical reaction depends on the size of the air bubbles produced in the bubbler, the interaction of contaminant with reagent

molecules, the rapidity of the reaction, and a sufficient excess of reagent solution. If the reaction is rapid and a sufficient excess of reagent is maintained in the liquid, complete retention of the contaminant is achieved regardless of the volume of air sampled. If the reaction is slow and the sampling rate is not low enough, collection efficiency will suffer.

Four basic absorbers used for the collection of gases and vapors are 1) simple gas washing bottles, 2) spiral and helical absorbers, 3) fritted bubblers, and 4) glass-bead columns. Sampling and absorbent capacities of these absorbers are found on Table S-2. Their function is to provide sufficient contact between the contaminant in the air and the absorbing liquid.

Petri, Dreschsel, and midget impingers are examples of simple gas washing bottles. They function by applying a suction to an outlet tube which causes sample air to be drawn through an inlet tube into the lower portion of the liquids contained in these absorbers. They are suitable for collecting nonreactive gases and vapors that are highly soluble in the absorbing liquid where they form a near perfect solution. The absorption of methanol and butanol in water, esters in alcohol, and organic chlorides in butyl alcohol are examples. They are also used for collecting gases and vapors that react rapidly with a reagent in the sampling media. High collection efficiency is achieved, for example, when toluene diisocyanate is hydrolyzed to toluene diamine in Marcali⁽²¹⁾ solution. Hydrogen sulfide reaction with cadmium sulfate and ammonia neutralized by dilute sulfuric acid are other examples.

Several methods for testing the efficiency of an absorbing device are available: 1) by series testing where enough samplers are arranged in series so that the last sampler does not recover any of the test gas or vapor; 2) by sampling from a dynamic standard atmosphere or from a gas-tight chamber or tank containing a known gas or vapor concentration; 3) by comparing results obtained with a device known to be accurate; and 4) by introducing a known amount of gas or vapor into a sampling train containing the absorber being tested.

Cold Traps

Cold traps are used for collecting materials in liquid or solid form primarily for identification purposes.

TABLE S-2. Sampling Rate and Absorbent Capacity of Absorbers⁽⁶⁾

| Type of Absorber | Absorbent Capacity (ml) | Sample Rate ml/min |
|----------------------------|-------------------------|--------------------|
| Simple gas washing bottles | 5-100 | 5-3000 |
| Spiral and helical | 10-100 | 40-500 |
| Fritted bubblers | 1-100 | 500-100,000 |
| Glass-bead column | 5-50 | 500-2000 |

Vapor is separated from air by passing it through a coil immersed in a cooling system, i.e., dry ice and acetone, liquid air, or liquid nitrogen. These devices are employed when it is difficult to collect samples efficiently by other techniques. Water is extracted along with organic materials and two-phase systems result.

Plastic Sampling Bags

Plastic bags (as used for grab sampling) can also be used for collecting integrated air samples. Samples can be collected for eight hours, at specific times during the day, or over a period of several days. The bags may be mounted on workers as personal samplers or may be located in designated areas unattended.

Solid Adsorbents

Activated Charcoal

Charcoal is an amorphous form of carbon formed by burning wood, nutshells, animal bones, and other carbonaceous materials. A wide variety of charcoals are available; some are more suitable for liquid purification, some for decolorization, and others for air purification and air sampling.

Ordinary charcoal becomes activated charcoal by heating it with steam to 800°-900°C. During this treatment, a porous, submicroscopic internal structure is formed which gives it an extensive internal surface area, as large as 1000 m² per gram of charcoal, which greatly enhances its adsorption capacity.

Activated charcoal is an excellent adsorbent for most organic vapors. During the 1930s and 1940s, it was used in the then well-known activated charcoal apparatus⁽²²⁾ for the collection and analysis of solvent vapor. The quantity of vapor in the air sample was determined by a gain in weight of the charcoal tube. Lack of specificity, accuracy, and sensitivity of the analysis and the difficult task of equilibrating the charcoal tube, however, discouraged use.

Renewed interest in activated charcoal as an adsorbent for sampling organic vapors appeared in the 1960s.⁽²³⁻²⁵⁾ The ease with which carbon disulfide extracts organic vapors from activated charcoal and the capability of microanalysis by gas chromatography are the reasons for its current popularity. Today, air sampling procedures using activated charcoal are widely used by industrial hygienists⁽²⁶⁻²⁹⁾ and form the basis of the majority of the official analytical methods for organic materials recommended by the National Institute for Occupational Safety and Health (NIOSH) and the Occupational Safety and Health Administration (OSHA).^(1,2)

Analytical information on selected NIOSH procedures is given in Table S-3 and is based on the extensive evaluation of the charcoal tube procedure by NIOSH.⁽³⁰⁾ This study showed that the charcoal tube method is generally adequate for hydrocarbons, chlorinated

TABLE S-3. Collection and Analysis of Gases and Vapors (Adsorption on Charcoal)

| Gas or Vapor | Sample Volume (L) | Useful Range (ppm) | Desorption | References |
|----------------------|-------------------|--------------------|-----------------------------------|-------------|
| Allyl alcohol | 10 | 0.2-8 | CS ₂ , 5% 2-propanol | NIOSH S-52 |
| Amyl acetate | 10 | 12.5-350 | CS ₂ | NIOSH S-51 |
| Butadiene | 1 | 100-3000 | CS ₂ | NIOSH S-91 |
| Butyl acetate | 10 | 20-600 | CS ₂ | NIOSH S-46 |
| Butyl alcohol | 10 | 10-300 | CS ₂ , 1% 2-propanol | NIOSH S-66 |
| Butyl Cellosolve | 10 | 5-150 | 5% methanol in methylene chloride | NIOSH S-76 |
| Camphor | 20 | 0.2-35 | CS ₂ , 1% methanol | NIOSH S-10 |
| Cumene | 10 | 5-150 | CS ₂ | NIOSH S-23 |
| Cyclohexanol | 10 | 5-150 | CS ₂ , 5% 2-propanol | NIOSH S-54 |
| Cyclohexane | 10 | 30-900 | CS ₂ | NIOSH S-28 |
| Diacetone alcohol | 10 | 5-150 | CS ₂ , 5% 2-propanol | NIOSH S-55 |
| Dioxane | 10 | 10-300 | CS ₂ | NIOSH S-360 |
| Ethyl acetate | 6 | 40-1200 | CS ₂ | NIOSH S-49 |
| Ethyl acrylate | 10 | 2.5-75 | CS ₂ | NIOSH S-35 |
| Ethyl alcohol | 1 | 100-3000 | CS ₂ , 1% 2-butanol | NIOSH S-56 |
| Ethyl ether | 3 | 40-1200 | Ethyl acetate | NIOSH S-80 |
| Glycidol | 50 | 5-1500 | Tetrahydrofuran | NIOSH S-70 |
| Heptane | 4 | 50-1500 | CS ₂ | NIOSH S-89 |
| Hexane | 4 | 50-1500 | CS ₂ | NIOSH S-90 |
| Isopropyl alcohol | 3 | 40-1000 | CS ₂ , 1% 2-butanol | NIOSH S-65 |
| Mesityl oxide | 10 | 2.5-75 | CS ₂ , 1% methanol | NIOSH S-12 |
| Methyl acetate | 7 | 20-500 | CS ₂ | NIOSH S-42 |
| Petroleum distillate | 4 | 50-1500 | CS ₂ | NIOSH S-380 |
| Propyl alcohol | 10 | 20-400 | CS ₂ , 1% 2-propanol | NIOSH S-62 |
| Stoddard solvent | 3 | 50-1500 | CS ₂ | NIOSH S-382 |
| Styrene | 5 | 20-600 | CS ₂ | NIOSH S-30 |

hydrocarbons, esters, ethers, alcohols, ketones, and glycol ethers that are commonly used as industrial solvents. Compounds with low vapor pressure and reactive compounds (e.g., amines, phenols, nitrocompounds, aldehydes, and anhydrides) generally have low desorption efficiencies from charcoal and require alternative sorbents such as silica gel, porous polymers, or reagent systems for collection.

Inorganic compounds, such as ozone, nitrogen dioxide, chlorine, hydrogen sulfide, and sulfur dioxide, react chemically with activated charcoal and cannot be collected for analysis by this method.

Even for substances recommended for sampling on charcoal, this sorbent may not always be ideal. Reference to Table S-3 will indicate that carbon disulfide is the recommended desorption solvent for nonpolar compounds while a variety of desorption cocktails are required for the more polar compounds. Difficulties arise, therefore, when sampling mixtures of polar and nonpolar compounds as each will give poor recoveries with the other's desorption solvent. Several alternative, more universal, solvents have been investigated,⁽³¹⁻³³⁾ but none of these has achieved wide recognition. In

such circumstances, it may be necessary to take two samples and desorb twice.

The volume of air that can be collected without loss of contaminant depends on the sampling rate, sampling time, volatility of the contaminant, and concentration of contaminant in the workroom air. For many organic vapors, a sample volume of 10 L (1.0 L/min) can be collected without significant loss in NIOSH-recommended tubes. A breakthrough of more than 20% into the backup section indicates that some of the sample was lost. Optimum sample volumes are found in the NIOSH procedures.

The sample volume for gases and highly volatile solvents must necessarily be smaller. A 3% breakthrough was found to occur on NIOSH-recommended tubes at 0.2 L/min for 15 minutes in an environment containing 5 ppm of vinyl chloride. Losses occurred before 5 L of the sample were collected in a 200 ppm vinyl chloride environment at a sampling rate of 0.05 L/min.⁽²⁹⁾

It is always best to refer to an established procedure for proper sampling rates and air sample volumes. In the absence of such information, breakthrough ex-

periments must be performed before field sampling is attempted. The concentration of contaminant expected to be found in the field should be prepared in a sampling jar or fume chamber and tests should be made on it. See Chapter F for the preparation of known concentrations.

After the procedure has been checked out, field sampling may be performed. Immediately before sampling, the ends of the charcoal tube are broken, rubber or Tygon® tubing is connected to the backup end of the charcoal tube, and air is drawn through the sampling train with a calibrated battery or electrically driven suction pump. A personal or area sample may be collected. The duration of the sampling may be several minutes or up to eight hours depending on the information desired. In any case, air flow should be checked periodically with a flowmeter while the sampling is in progress. Afterwards, when sampling is completed, plastic caps or masking tape (but not rubber caps) are placed on the ends of the tube.

For each new batch of charcoal tubes, the analysis blank, the aging, collection efficiency, and recovery characteristics for a given contaminant must be determined. This may be achieved by introducing a known amount of the contaminant into a freshly opened charcoal tube, passing clean air through it to simulate sampling conditions, and carrying through its analysis with the field samples. Another charcoal tube, not used to sample, is opened in the field and used as a field blank.

The first step in the analysis procedure is to remove the contaminant from the charcoal. An early drawback to using charcoal for air sampling was the difficulty in recovering samples for analysis. Steam distillation was only partially effective. Thermal desorption and extraction with carbon disulfide have been found, in many instances, to be quite satisfactory. Thermal desorption of vinyl chloride, methyl chloride, and vinylidene chloride was achieved by placing the charcoal sample in a special tube muffle furnace and purging it with nitrogen. The expelled vapors were introduced directly into a gas chromatograph.⁽²⁸⁾

The most frequently used desorbant is carbon disulfide. Unfortunately, carbon disulfide does not always completely remove the sample from charcoal. Recovery varies for each contaminant and batch of charcoal used. The extent of individual recoveries must be determined experimentally and a correction for desorption efficiency applied to the analytical result.⁽²³⁾ Over a narrow range of analyte concentrations, as used in the NIOSH validations,⁽³⁰⁾ this desorption efficiency is essentially constant, but it may vary widely over larger concentration ranges, particularly for polar compounds.⁽³⁴⁾ Desorption efficiency can also be affected by the presence of water vapor and of other contaminants.⁽³⁵⁾ NIOSH⁽¹⁾ recommends that methods should be used only where

the desorption efficiency is greater than 75%; ideally, it should be greater than 90%.

The practical desorption step in charcoal analysis is also critical since, upon the addition of carbon disulfide to charcoal, the initial heat of reaction may drive off the more volatile components of the sample. This can be minimized by adding charcoal slowly to precooled carbon disulfide. Another technique is to transfer the charcoal sample to vials lined with Teflon septum caps and to introduce the carbon disulfide with an injection needle. The sealed vial will prevent the loss of any volatilized sample.

It should be emphasized that carbon disulfide is a highly toxic solvent that produces serious effects on the cardiovascular and nervous systems. Care should be exercised in handling the solvent, and the analytical procedure should be performed in a well ventilated area.

Several quality assurance schemes have been developed which apply to the charcoal tube method. One of these is the Proficiency Analytical Testing (PAT) Program⁽³⁶⁾ and the Laboratory Accreditation Program of the American Industrial Hygiene Association (AIHA).⁽³⁷⁾ Another is the Health and Safety Executive (HSE) Workplace Analysis Scheme for Proficiency (WASP). Details of these programs may be obtained from The Laboratory Accreditation Coordinator, AIHA, 345 White Pond Drive, Akron, Ohio 45320; and the WASP Coordinator, HSE, Occupational Medicine and Hygiene Laboratories, London NW2 6LN, United Kingdom.

Silica Gel

Silica gel is an amorphous form of silica derived from the interaction of sodium silicate and sulfuric acid. It has several advantages over activated charcoal for sampling gases and vapors: 1) polar contaminants are more easily removed from the adsorbent by a variety of common solvents, 2) the extractant does not usually interfere with wet chemical or instrumental analyses, 3) amines and some inorganic substances for which charcoal is unsuitable can be collected, and 4) the use of highly toxic carbon disulfide is avoided.

One disadvantage of silica gel is that it will adsorb water. Silica gel is electrically polar, and polar substances are preferentially attracted to active sites on its surface. Water is highly polar and is tenaciously held. If enough moisture is present in the air or if sampling is continued long enough, water will displace organic solvents, which are relatively nonpolar in comparison, from the silica gel surface. With water vapor at the head of the list, compounds in descending order of polarizability are alcohols, aldehydes, ketones, esters, aromatic hydrocarbons, olefins, and paraffins. It is obvious, then, that the volume of moisturized air that can be effectively passed over silica gel is limited.

In spite of this limitation, silica gel has proven to be

an effective adsorbent for collecting many gases and vapors. Even under conditions of 90% humidity, relatively high concentrations of benzene, toluene, and trichloroethylene are quantitatively adsorbed on 10 g of silica gel from air samples collected at the rate of 2.5 L/min for periods of at least 20 minutes or longer.^(38,39) Under normal conditions, hydrocarbon mixtures⁽⁴⁰⁾ of 2 to 5 carbon paraffins, low molecular weight sulfur compounds (H₂S, SO₂, mercaptans), and olefins concentrate on silica gel at dry ice-acetone temperature if the sample volume does not exceed 10 L. Significant losses of ethylene, methane, ethane, and other light hydrocarbons occur if sampling volume is extended to 30 L.

More recent usage, however, has concentrated on smaller sample tubes (in similar sizes to the NIOSH range of charcoal tubes) operated at room temperature. NIOSH⁽¹⁾ recommends such tubes for a variety of more polar chemicals such as amines, phenols, amides, and inorganic acids.

Much the same considerations apply to silica gel tubes as to the charcoal tubes; the sampling capacity and desorption efficiency for the compound of interest should be determined before use, or a reliable officially established method should be used. A variety of desorption solvents will be needed for desorbing specific compounds with high efficiency; polar desorption solvents, such as water or methanol, are commonly applied.

Thermal Desorption

Because of the high toxicity and flammability of carbon disulfide and the labor intensive nature of the solvent desorption procedure, a useful alternative is to desorb the collected analyte thermally.⁽⁴¹⁻⁴³⁾ Except in a few cases, this is not practical with charcoal as adsorbent since the temperature needed for desorption (e.g., 300°C) would result in some decomposition of the analytes. Carbon molecular sieves or, more frequently, porous polymer adsorbents, in particular Tenax, Porapak Q and Chromosorb 106, are used instead. Of these, Tenax has the lowest thermal desorption blank (typically less than 0.1 µg/g of adsorbent, when properly conditioned) but only modest adsorption capacity compared with carbon.

The thermal desorption procedure typically uses larger tubes than the NIOSH method; usually 200–500 mg of sorbent are used, depending on type. Desorption can be made fully automatic, and analysis is usually carried out by gas chromatography. Some desorbers also allow automatic selection of sample tubes from a multiple-sample carousel. The whole sample can be transferred to the gas chromatograph, resulting in greatly increased sensitivity compared with the solvent desorption method. Alternatively, some desorbers allow the desorbed sample to be held in a reservoir from

which aliquots are withdrawn for analysis, but then the concentrating advantage is reduced.

Thermal desorption has been adopted as a (nonexclusive) recommended method in the U.K.,⁽³⁾ Germany,⁽⁴⁾ and the Netherlands,⁽⁴⁴⁾ but it is less widely accepted in the U.S. NIOSH⁽¹⁾ has only two methods based on thermal desorption (P&CAM 252/299 and P&CAM 213); both are proposed (E) methods and do not appear in the 3rd Edition. The U.S. Environmental Protection Agency (EPA),⁽⁴⁵⁾ however, has a number of methods based on thermal desorption (and mass spectrometry), reflecting a greater interest in the method for environmental, as opposed to workplace, monitoring.

The main disadvantage of thermal desorption directly with an analyzer is that it is essentially a "one-shot" technique; normally, the whole sample is analyzed. This is why many such methods are linked to mass spectrometry. However, with capillary chromatography, it is usually possible to split the desorbed sample before analysis and, if desired, the vented split can be collected and reanalyzed.⁽⁴⁶⁾ Alternatively, the desorbate can be split between two capillary columns of differing polarity.⁽⁴⁷⁾

Desorption efficiency is usually 100% for the majority of common solvents and similar compounds in a boiling range of approximately 50° to 250° C. Thus, the analysis of complex mixtures is easier than for charcoal or silica gel solvent desorption methods although, if a wide boiling range is to be covered, more than one sorbent may be required. Thus, gasoline may be monitored by a Chromosorb 106 tube and carbon tube in series.⁽⁴⁸⁾ An extensive list of recommended sampling volumes and minimum desorption temperatures for Tenax is given in Brown and Purnell.⁽⁴³⁾

Sampling Train

Except for grab samplers (described above) and diffusive samplers (described below), sampling devices are used in conjunction with a sampling pump and air metering device. To avoid contaminating the metering device, it is usually placed downstream of the sampler during the sampling period, but since many samplers introduce back-pressure, the sampling train should be precalibrated by the use of an external flowmeter upstream of the sampling head. Air movers are described in *Chapter L* and calibration in *Chapter F*.

Analysis of Gases and Vapors

No attempt is made to provide a complete list of analytical procedures. Table S-3 provides pertinent analytical information for the collection of a number of organic vapors on charcoal tubes, as found in the *NIOSH Manual*.⁽¹⁾ Table S-4 lists some gases and vapors that may be analyzed by wet chemical methods or by ultraviolet spectrophotometry; it is a selection from two

primary compendia of methods, the AIHA Analytical Chemistry Committee⁽⁴⁹⁾ and the Intersociety Committee.⁽⁶⁾ Source references are given in these compendia. Other useful sources are Hansen,⁽⁵⁰⁾ Jacobs,⁽⁵¹⁻⁵²⁾ the "Methods for Detection of Toxic Substances in Air" series,⁽⁵³⁾ Ruch,^(54,55) and Thomas.⁽⁵⁶⁾ Spectrophotometric methods have now been replaced largely by direct-reading instruments or detector tubes (*Chapter T*) or by High Performance Liquid Chromatography (HPLC) or other instrumental techniques.^(1,2) However, they still have their place, particularly for highly reactive species such as isocyanates⁽²¹⁾ and formaldehyde.⁽⁵⁷⁾

Calculations

The collected sample is analyzed, either directly if a gas phase or impinger sample or after desorption if collected on a solid sorbent, using appropriate gas or liquid standard solutions to calibrate the analytical instrument. Gas phase samples give a result directly in

ppm (v/v), but other types of samples will give a mass of analyte per collected sample, or a concentration, which can be converted to a mass by multiplying by the sample volume.

The mass concentration of the analyte in the air sample is then calculated using the following equations.

Impinger

$$C = \frac{m - m_{\text{blank}}}{SE \times V} \quad (2)$$

where: C = mass concentration of analyte in air (mg/m³)

m = mass of analyte in sample (μg)

m_{blank} = mass of analyte in blank (μg)

SE = sampling efficiency

V = volume of air sampled (liters)

TABLE S-4. Some Colorimetric Procedures for Gases and Vapors

| Gas or Vapor | Sampler | Sorbent | Analysis | Reference |
|--------------------------------|-------------|------------------------------------|--|-----------|
| Acetaldehyde | Bubbler | Water | Iodoform reaction | 49 |
| Acetates | Bubbler | Ethanol | Hydroxamic acid | 49 |
| Acetic acid | Wash bottle | Glycerol/water | pH change | 49 |
| Acetonitrile | Syringe | Permanganate | Color change | 49 |
| Acrolein | Bubbler | Hexylresorcinol | Spectrophotometry | 6 |
| Aldehydes | Bubbler | MBTH | Spectrophotometry | 6 |
| Amines | Bubbler | HCl in isopropanol | Ninhydrin/spectrophotometry | 6, 49 |
| Ammonia | Bubbler | Dil H ₂ SO ₄ | Phenol/hypochlorite spectrophotometry | 6 |
| Aniline | Bubbler | Dil H ₂ SO ₄ | Spectrophotometry | 49 |
| Benzene | U-tube | Silica gel | Spectrophotometry | 49 |
| Butanol | Bubbler | Water | Chromate oxidation | 49 |
| Carbon disulfide | Glass beads | Copper/diethylamine | Color reaction | 49 |
| Chlorine | Bubbler | Methyl orange | Spectrophotometry | 6 |
| Ethanol | Impinger | Water | Chromate oxidation | 49 |
| Formaldehyde | Wash bottle | Water | Chromotropic acid | 6 |
| Formaldehyde | Impinger | Bisulfite | Iodine titration | 49 |
| Hydrogen sulfide | Bubbler | Iodine solution | Iodine oxidation | 49 |
| Mercaptans | Bubbler | Mercuric acetate | Phenylenediamine/spectrophotometry | 6 |
| Methanol | Impinger | Water | Fuchsin/formaldehyde | 49 |
| Methyl ethyl ketone | U-tube | Silica gel | Iodoform reaction | 49 |
| Methylene bisphenyl isocyanate | Impinger | Acid | Diazotation/coupling/spectrophotometry | 49 |
| Nitrobenzene | Bubbler | Ethanol | Spectrophotometry | 49 |
| Nitrogen dioxide | Bubbler | Naphthylethylenediamine | Color reaction | 6, 49 |
| Nitromethane | Bubbler | Phosphate | Diazo coupling/spectrophotometry | 49 |
| Ozone | Impinger | KI | Titration | 49 |
| Phenol | Impinger | Ethanol | Spectrophotometry | 49 |
| Phenol | Impinger | NaOH solution | Aminoantipyrene/spectrophotometry | 6 |
| Pyridine | Bubbler | Ethanol | Spectrophotometry | 49 |
| Sulfur dioxide | Impinger | Tetrachloromercurate | Spectrophotometry | 6, 49 |
| Toluene | U-tube | Silica gel | Spectrophotometry | 49 |
| Toluene diisocyanate | Impinger | Acid | Diazotation/coupling/spectrophotometry | 49 |

Absorbent Tube

$$C = \frac{m_1 + m_2 - m_{\text{blank}}}{DE \times V} \quad (3)$$

where: m_1 = mass of analyte on first tube section (μg)
 m_2 = mass of analyte on backup tube section (if used) (μg)
 DE = desorption efficiency corresponding to m_1

Note: If it is desired to express concentrations reduced to specified conditions, e.g., 25°C and 101 kPa, then,

$$C_{\text{corr}} = C \left(\frac{101}{P} \right) \left(\frac{T}{298} \right) \quad (4)$$

where: P = actual pressure of air sampled (kPa)
 T = absolute temperature of air sampled ($^{\circ}\text{K}$).

Volume Fraction

The volume fraction of the analyte in air, in ppm (v/v), is

$$C' = C_{\text{corr}} \left(\frac{24.5}{MW} \right) \quad (5)$$

where: MW = molecular mass of the analyte of interest (g/mol).

Diffusive Samplers**Overview**

A diffusive sampler is a device which is capable of taking samples of gas or vapor pollutants from the atmosphere at a rate controlled by a physical process, such as diffusion through a static air layer or permeation through a membrane, but which does not involve the active movement of the air through the sampler.⁽⁵⁸⁾ It should be noted that in the U.S., the adjective “passive” is preferred in describing these samplers and should be regarded as synonymous with “diffusive.”

This type of diffusive sampler should not be confused with the annular or aerosol denuders, which not only rely on diffusion to collect the gas or vapors, but also upon the air in question being simultaneously drawn through the annular inlet into the sampler. Aerosol particles have diffusion coefficients too low to be collected on the annular inlet and are trapped on a backup filter. More information on denuders can be found in *Chapter R*.

Diffusive sampling in the occupational environment dates back at least to the 1930s when qualitative devices were described, but the first serious attempt to apply science to quantitative diffusive sampling was in 1973 when Palmes described a tube-form sampler for nitrogen dioxide.⁽⁵⁹⁾ Since then, a wide variety of samplers have been described, some relying on diffusion through an air-gap, some relying on permeation through a

membrane, and some using both techniques, for the rate-controlling process in sampling.⁽⁵⁹⁻⁶²⁾ Many of these devices are commercially available.

The theoretical basis for diffusive sampling is now well established.⁽⁵⁸⁾ Diffusion and permeation processes can both be described in derivations of Fick's first law of diffusion (Equation 6) which result in expressions relating the mass uptake by the sampler to the concentration gradient, the time of exposure, and the sampler area exposed to the pollutant atmosphere.⁽⁶³⁾ Expressions have also been derived for the application of Fick's law to diffusive sampling in the “real” world, i.e., taking into account nonsteady-state sampling, the effects of fluctuating concentrations, sorbent saturation, wind velocity and turbulence at the sampler surface, temperature, pressure, and so on.⁽⁶⁴⁻⁶⁵⁾ Except for sorbent saturation, which may lead to reduced (although sometimes predictable) uptake rates, these modifications to the basic Fick's law expression do not lead to significant errors for well-designed samplers. Such samplers may be regarded as truly integrating devices with accuracies similar to those of active samplers.

A variety of diffusive samplers have been described⁽⁶⁶⁾ and only a selection of the major types manufactured can be described here. Diffusive equivalents to the more familiar pumped methods exist for nearly all types; the main exception being the direct collection of gas samples, where the nearest equivalent is an evacuated canister. Thus, the diffusive equivalent of an impinger is a liquid-filled badge such as the Pro-Tek™ inorganic monitor or the SKC badge; the diffusive equivalent of the charcoal tube is the charcoal badge such as the 3M OVM or the MSA VaporGard™ organic; and the diffusive equivalent of the thermal desorption method is the Perkin-Elmer tube or the SKC thermal desorption badge. There are also diffusive devices based on reagent-impregnated solid supports, but these are mostly direct reading and are dealt with in *Chapter T*.

In general, the regulatory authorities have been reluctant to accept diffusive monitoring methods, except in the U.K. and the Netherlands where several such methods have been adopted as nonexclusive recommended methods.^(3,44) However, the Luxembourg Symposium⁽⁵⁸⁾ concluded that:

- The theoretical basis for diffusive sampling has been confirmed by laboratory and field trials.
- Active and diffusive sampling are complementary approaches, having areas of applicability which may overlap. Each has its role in a strategy for monitoring worker exposure.
- In general, there seems to be no significant difference between the accuracy and precision of diffusive sampling and those of other monitoring systems such as active pumped sampling.

- It was agreed that, as a general principle, any method is acceptable by regulatory authorities and hygienists if used by experts within its defined limitations. This applies equally to diffusive samplers.

The symposium also concluded that validation of all sampling systems is essential both in the laboratory and in the field. It also recommended that an established evaluation protocol be followed such as the NIOSH⁽⁶⁷⁾ or the HSE.⁽⁶⁸⁾

Calibration

The basic expression of Fick's Law is

$$J = \frac{D(C_o - C_e)}{L} \quad (6)$$

and

$$Q = (DA) \left(\frac{C_o - C_e}{L} \right) t \quad (7)$$

where: J = diffusive flux (g/cm²-sec)
 D = coefficient of diffusion (cm²/s)
 A = cross-sectional area of diffusion path (cm²)
 L = length of diffusion path (cm)
 C_o = external concentration being sampled (g/cm³)
 C_e = concentration at the interface of the sorbent (g/cm³)
 Q = mass uptake (g)
 t = sampling time (sec)

It is apparent from an inspection of these equations that the expression DA/L has units of cm³/s and therefore represents what can be considered as a "sampling rate" of the diffusive sampler when compared to a pumped sampling system. This simple use of the sampling rate concept has been of considerable value to users of the devices and is often expressed in the dimensionally equivalent units of ml/min. Knowledge of the geometry of the sampler (which will be fixed for any given sampler type) permits the calculation of the sampling rate provided the diffusion coefficient is known. A number of manufacturers have published tables of sampling rates calculated in this way, most of whom have used the same source of published diffusion coefficients.⁽⁶⁹⁾ Diffusion coefficients that are not in this list can be calculated theoretically.⁽⁷⁰⁾ Representative sampling rates, supplied by DuPont and 3M, range from 20 to 45 cc/min (see Table SI-1).

Environmental Factors Affecting Monitor Performance^(58,65-68)

Temperature and Pressure

From Maxwell's equation, the diffusion coefficient, D, is a function of absolute temperature, T, and pressure, P:

$$D = f(T^{3/2}, P^{-1}) \quad (8)$$

But from the general gas law;

$$PV = nRT \quad (9)$$

$$C = \frac{n}{V} = \frac{P}{RT}$$

Substituting Equations 8 and 9 in Equation 7, we get:

$$Q = f \left(\frac{P}{T}, \frac{T^{3/2}}{P} \right) = f(T)^{1/2} \quad (10)$$

Thus, Q is independent of pressure, P, but dependent on the square root of absolute temperature, T. In practice, the temperature dependence of the sampling rate at ambient temperature levels (about 0.2% per °C) may be ignored. However, temperature may affect the absorption/adsorption capacity of a sorbent adversely.

Humidity

High humidity can affect charcoal adsorption adversely, resulting in a reduction in saturation capacity for charcoal badges. If the sampler becomes saturated, C_e in Equation 5 is no longer zero, and the sampling rate becomes nonlinear. Porous polymers used for thermal desorption are relatively unaffected by humidity.

Transients

Simple derivations of Fick's Law assume steady-state conditions, but in the practical use of such samplers, the ambient concentrations of pollutants are likely to vary widely. The question then arises whether a diffusive sampler will give a truly integrated response or will "miss" short-lived transients before they have had a chance to diffuse into the sampler. The problem has been discussed theoretically^(64,71,72) and practically.^(73,74) Generally, transients do not present a significant problem provided the *total* sampling time is well in excess of the time constant of the sampler, i.e., the time a molecule takes to diffuse into the sampler under steady-state conditions. The time constant of most commercial samplers is between 1 and 10 sec.

Sorbent Factors

All diffusive samplers rely on sorbents having a high affinity for the contaminant being sampled, i.e., C_e = zero in Equation 6, and uptake is linearly proportional to concentration and time of exposure. Useful checks on sorbent suitability are a back-diffusion test given in Bartley⁽⁷⁵⁾ and the measurement of adsorption isotherms.⁽⁷⁶⁾

Face Velocity

Diffusion samplers also rely on the external concentration, i.e., C_o in Equation 6, being maintained at the sampler surface. In the absence of sufficient air move-

ment across the face of the sampler, transport of pollutant to the surface may itself be limited by diffusion and the effective sampling rate will be reduced. At the other extreme, very high air velocities may induce turbulence within the sampler body if the draught shield is inadequate; the effective diffusion path length will be reduced and the sampling rate increased. The magnitude of these effects will vary with the geometry and design of particular samplers, although for the majority of modern samplers, sampling rates are reasonably constant within the range of air velocities likely to be encountered in workplace personal monitoring. Samplers with a large surface area ("badge" types) should not be used in "static" positions where air velocities may be below their critical values for this type of samplers (about 0.2 m/s).

Calculations

The method of calculation of atmospheric concentrations is essentially the same as for pumped samplers, i.e., the collected sample is analyzed and the total weight of analyte on the sampler determined. Then, as before,

$$C = \frac{m_1 + m_2 - m_{\text{blank}}}{DE \times V} \quad (3)$$

(m_2 and DE are ignored for liquid sorbent badges)

V , the total sample volume, is calculated from the effective sampling rate (L/min) and the time of exposure (min).

This calculation gives C in mg/m^3 ; strictly speaking, an appropriate sampling rate for the ambient temperature and pressure should be made as Equation 9 assumes C is in ppm.

Alternatively, sampling rates can be expressed in units such as $\text{ng}/\text{ppm}/\text{min}$ (dimensionally equivalent to cm^3/min), when C' is calculated directly in ppm:

$$C' = \frac{m_1 + m_2 - m_{\text{blank}}}{DE \times U \times t'} \times 100 \quad (11)$$

where: U = sampling rate ($\text{ng}/\text{ppm}/\text{min}$)

t' = sampling time (min)

Note: m_2 is relevant only to samplers with a backup section, and an additional multiplication factor may be needed to account for differing diffusion path lengths to primary and backup sections.

Types of Monitors

Diffusive samplers are available for both organic and inorganic species. Most organic monitors use activated charcoal as the collection medium. Both diffusion and permeation devices are available. As a general rule, organic badges can be used to monitor any compound that can be sampled by charcoal tube-pump methods. Each monitor has a unique design, and the operation characteristics will be discussed in detail in the fol-

lowing section. As a further note, all of these require gas chromatographic analysis for determination of the contaminant concentration.

Diffusion monitors for inorganic gases and vapors are far more diverse in design and more chemispecific than the more generally absorbing organic monitors. There are also many direct-reading, passive monitors for inorganic contaminants (see *Chapter T*).

Accuracy of Diffusive Monitoring

The overall accuracy of diffusive monitors has been studied extensively.⁽⁵⁶⁾ Most of the devices available commercially meet NIOSH and OSHA standards. NIOSH recommends that monitors produce results of $\pm 25\%$ for 95% of the samples tested in the range of 0.5 to 2.0 times the environmental standard. OSHA's accuracy requirement varies from $\pm 25\%$ to $\pm 50\%$, depending on the individual standard.

Field and laboratory test results on several commercially available badges are found in the literature. Brown,⁽⁷⁷⁾ for example, examined the Perkin-Elmer tube for acrylonitrile, benzene, butadiene, carbon disulfide, and styrene and found the sampler to be at least as accurate as the equivalent pumped method. Laboratory precision was, on the average, 10% for the diffusive sampler. Field precision was 12% for the diffusive sampler and 13% for the pumped sampler.

Kennedy⁽⁷⁸⁾ evaluated a range of inorganic samplers, including 3M, DuPont, MSA, REAL, and SKC samplers, and found they generally met NIOSH criteria.

A European interlaboratory comparison⁽⁷⁹⁾ of the 3M badge exposed to butanol, pentanal, trichloroethane, octane, butyl acetate, 3-heptanone, xylene, α -pinene, and decane generally displayed good agreement with the charcoal tube. Exceptions were butanol and pentanal, where the diffusive samplers read low. Again, excluding butanol and pentanal, overall laboratory precision varied between 9% (xylene) and 13% (heptanone). The contribution of interlaboratory error was less than half of these values.

Lautenberger *et al.*⁽⁸⁰⁾ measured acrylonitrile, benzene, carbon tetrachloride, acetone, and toluene with DuPont's Pro-Tek G-AA Organic Vapor Air Monitoring Badges. In comparative testing, the badge demonstrated an overall accuracy at least equivalent to the charcoal method.

Kring *et al.*⁽⁸¹⁾ tested the DuPont Passive Colorimetric Air Monitoring Badge System for ammonia, sulfur dioxide, and nitrogen dioxide and found it met both NIOSH and OSHA accuracy requirements.

Seventy-eight pairs of side-by-side charcoal and 3M passive monitor samples were taken in the field and analyzed for 22 organic chemicals. The study⁽⁸²⁾ indicated that the monitor assayed concentrations of 20 of these chemicals equally as well as charcoal tubes, on the basis of linear regression analysis.

Interpretation of Results

Once the analyses are completed, interpretation of results must be made. Federal laws require that exposure to gases and vapors shall not exceed OSHA permissible exposure limits published in 29 CFR 1910.1000 *et seq.* A comparison with these standards will establish if there is compliance with the law.

This mechanical approach to the problem should be modified to include a better understanding of the Threshold Limit Values or standards. For this purpose, the ACGIH TLV/BEI Committees publish a companion volume to its TLV/BEI booklet called *Documentation of the Threshold Limit Values and Biological Exposure Indices*.⁽⁸³⁾ NIOSH recommended exposure limits have also been published under the title of "NIOSH's Recommendations for Occupational Safety and Health Standards."⁽⁸⁴⁾ Other sources of information of this nature are the AIHA *Hygienic Guides* and the American National Standards Institute (ANSI) documents. On the other hand, measurements may be taken for reasons other than demonstrating compliance, e.g., an epidemiological survey, and an appropriate interpretation of results must be made.

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Instrument Descriptions

The instrument descriptions are grouped into two major categories: 1) gas and vapor collectors and 2) diffusion samplers and monitors. Instrument manufacturers' names, addresses, and telephone numbers are grouped in Tables SI-6 and SI-7. All tables appear at the end of the section.

Grab Samplers

S-1 Evacuated Flasks

Daco Products, Inc.
Alltech Associates, Inc.

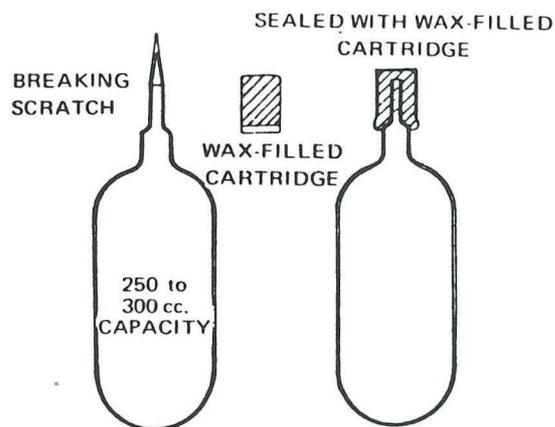
These are usually heavy-walled containers of 200, 500, or 1000 ml capacity. By means of a heavy-duty vacuum pump, the internal pressure is reduced (nominally)

to zero. Instrument S-1.a illustrates one such container. The neck of the container is drawn to a tip and sealed by heating during the final stages of evacuation. The sample is taken by breaking the sealed end. The barometric pressure and air temperature at the sampling site are noted. After sampling, the flask is resealed with a ball of wax, masking tape, or other suitable sealant and is sent to the laboratory for analysis.

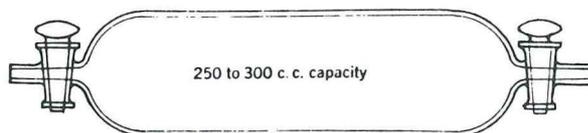
Instrument S-1.b illustrates a separatory flask fitted with glass stoppered cocks on each end. Alltech Associates supply 125-, 250-, and 500-ml gas sampling bulbs with septum ports. These tubes are suitable for partial evacuation. Evacuation is achieved by drawing a vacuum through one stem while the other is kept closed, then closing the open stem before the vacuum is turned off. These containers are available in glass, plastic, and metal.

Daco Products has a sampling system called "Chemist in the Can." It is a specially treated can under vacuum which is activated by pressing a button. The spent can containing the air sample is returned to the laboratory for analysis. The cans are reusable.

Alltech Associates also provides evacuated aerosol containers. These are of aluminum construction, 280 ml capacity, and measure 2.125 in. × 6 in. diameter. To fill the container, a hypodermic needle is inserted through a septum. A needle vacuum gauge is used to measure the vacuum level just before sampling and also during re-evacuation of the container.

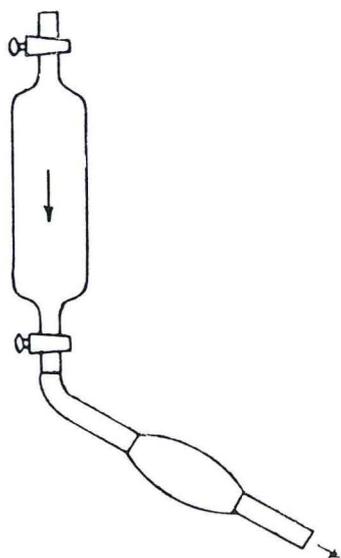


INSTRUMENT S-1.a. Evacuated sample container.⁽⁸⁵⁾

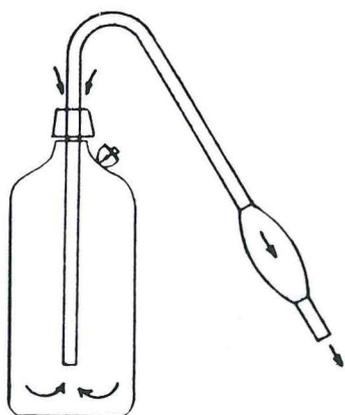


INSTRUMENT S-1.b. Gas or liquid displacement type sampling bottle.⁽⁸³⁾

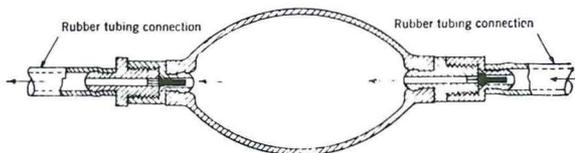
Except for the heavy-walled containers illustrated by Instrument S-1.a, no attempt is made to reduce the pressure to zero in other containers. However, the degree of evacuation must be known and is determined from the manometer pressure or vacuum gauge. This information, along with the barometric pressure and temperature at the sampling site, is used to calculate the actual volume of air or gas collected.



INSTRUMENT S-2.a. Filling container with rubber bulb hand aspirator.⁽⁸³⁾



INSTRUMENT S-2.b. Filling bottle with rubber bulb hand aspirator.⁽⁸³⁾

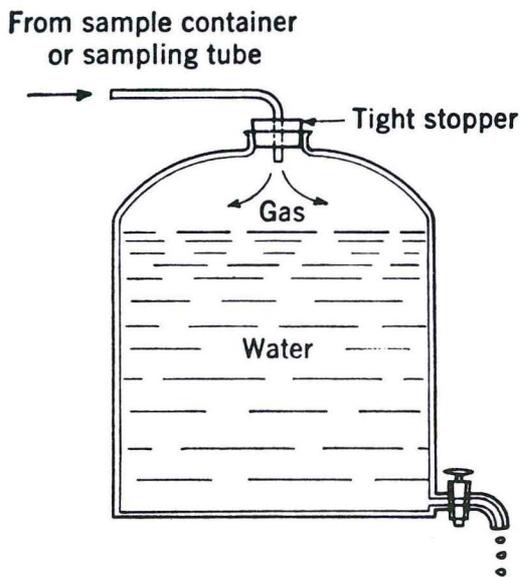


INSTRUMENT S-2.c. Rubber bulb hand aspirator.⁽⁸⁵⁾

S-2 Gas/Liquid Displacement Flasks

General Supply Houses (see Table SI-6)

Ordinary, sealable containers are used as gas/liquid displacement flasks. Many commercial firms sell suitable displacement flasks. A users' selection criteria should include required volume of sample to be taken, reactivity of the analyte of interest, and the practicality of using such a device (i.e., no pump is necessary with liquid displacement, but something must be done with the drained fluid). See Instruments S-2.a through S-2.d.

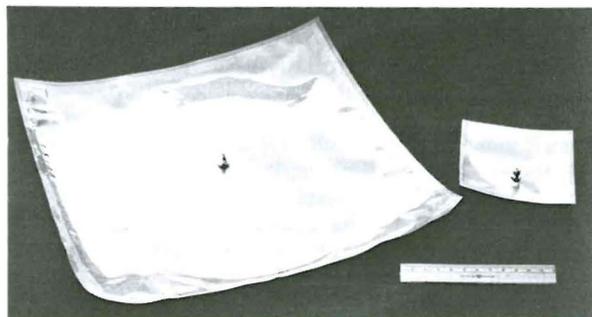


INSTRUMENT S-2.d. Aspirator bottle.⁽⁸⁵⁾

S-3 Flexible Plastic Containers (Sampling Bags)

- Alltech Associates, Inc.
- Anspec Company, Inc.
- Calibrated Instruments, Inc.
- Carborundum Plastics, Inc.

Sampling bags are widely used, available from a variety of manufacturers and distributors, made of a variety of materials, and come in a variety of sizes (from



INSTRUMENT S-3. Teflon sampling bag (Carborundum Plastics, Avondale, PA).

less than 1 L to 250 L). The materials include polyester (e.g., Aluminized Scotch Pak, Scotch Pak and Mylar®), polyvinylidene chloride (e.g., Saran®), and fluorocarbons (e.g., Chemton, Kel F, Aclor, Kynar®, Tedlar®, and Teflon®). Instrument S-3 illustrates a Teflon bag from Carborundum Plastics.

All bags should be leak tested, cleaned with compressed air, and conditioned before use. Three pump and cleaning cycles should be sufficient. The conditioning is first performed in the laboratory using test atmospheres. It is then repeated in the field before use by filling and emptying a bag several times at the sampling rate that will be used for taking the sample.

S-4 Hypodermic Syringes

General Supply Houses (see Table SI-6)

Syringes of 10 to 50 ml volume have been found satisfactory for air sampling.^(86,87) Suitable syringes should be gas-tight. They are available in glass and disposable plastic. Gas and vapor storage and decay curves for these devices must be determined. Advantages are their low cost, convenience, and ease of use.

Continuous Active Samplers

S-5 Bubblers and Gas Washing Bottles

Ace Glass, Inc.; BGI, Incorporated;
Corning Glass Works; Daco Products, Inc.;
Scientific Glass and Instruments Company

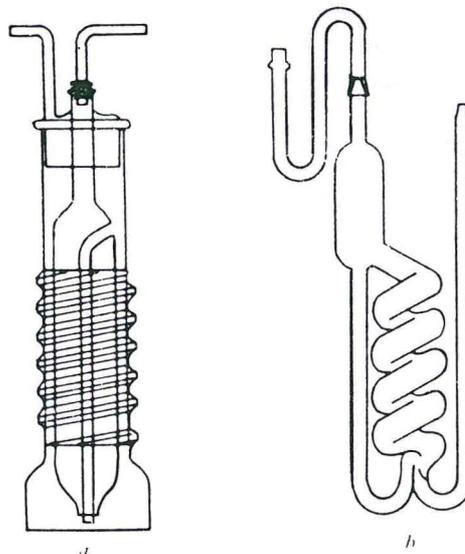
The midget impinger is the most widely used in this group and is illustrated in Instrument S-5.a; it is described in *Chapter P* (instrument descriptions P-43 and P-45). It is designed for impacting particles at a flow rate of 2.8 L/min; however, for industrial hygiene use as a bubbler, it is generally used with about 10 ml of absorbing solution and a flow rate of 1.0 L/min. No more than 20 ml of absorbing solution is added to the impinger flask. Air sampling is performed by connecting a personal pump or other source of suction to the outlet tube. The impinger is either hand-held or attached to worker's clothing. Care must be taken that the impinger does not tilt which could result in a loss of absorbing solution or reagent. Too much reagent solution or an excessive flow rate will also lead to loss of sample. Spill-proof impingers have been designed to minimize this problem and are commercially available (Daco Products).

Friedrichs and Milligan gas washing bottles are examples of spiral and helical absorbers (Instrument S-5.b). They may be used for collecting gaseous substances that are only moderately soluble in, or are slow in reacting with, reagents in the collection media. The spiral or helical structures provide for higher collection efficiency by allowing longer residence time of the contaminant within the tube. Slower acting and less



Model 9100

INSTRUMENT S-5.a. Midget impinger (Ace Glass, Inc.).

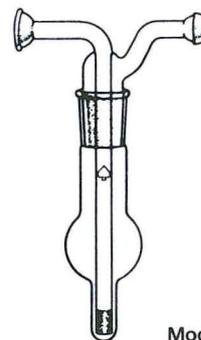


INSTRUMENT S-5.b. Spiral type absorbers.⁽⁶⁾



Model 9110

INSTRUMENT S-5.c. Midget gas bubbler (coarse frit) (Ace Glass, Inc.).



Model 9200

INSTRUMENT S-5.d. Nitrogen dioxide gas bubbler (Ace Glass, Inc.).

soluble substances are permitted more time to react with the absorbing solution.

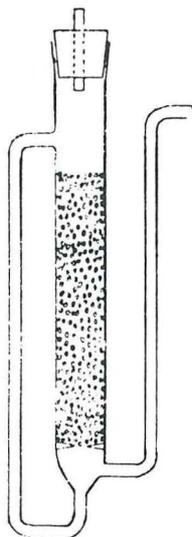
Gases and vapors that are sparingly soluble in the collecting medium may be sampled in fritted bubblers (Instruments S-5.c and S-5.d). They contain sintered or fritted glass or multiperforated plates at the inlet tube. Air drawn into these devices is broken up into very small bubbles, and the heavy froth that develops increases the contact of gas and liquid.

Frits come in various sizes and grades, usually designated as fine, medium, and coarse. A coarse frit is usually best for gases and vapors that are appreciably soluble or reactive. A medium porosity frit may be used for gases and vapors that are difficult to collect, but the sampling rate must be adjusted to maintain a flow of discrete bubbles. For highly volatile gaseous substances that are extremely difficult to collect, a frit of fine porosity may be required to break the air into extremely small bubbles and insure adequate collection efficiency. Air flow, however, must be controlled to avoid the formation of large bubbles by the coalescence of small bubbles. There is little value, for example, in using a fine porosity frit if air flow is increased and a large bubble population is produced. The finer the frit, however, the higher the pressure drop. Selection of proper frit should be made with all these factors in mind. The collection efficiency of the sampling equipment must be determined for specific contaminants involved.

S-6 Packed Glass-Bead Columns

General Supply Houses (see Table SI-6)

Packed glass-bead columns (Instrument S-6) are used for special situations where a concentrated solu-



INSTRUMENT S-6. Packed glass-bead column.⁽⁶⁾

tion is needed. Glass pearl beads are wetted with the absorbing solution and provide a large surface area for the collection of a sample. It is of historical interest to note that the absorption of benzene and other aromatic hydrocarbon vapors in nitrating acid has been performed with this type of absorber. It is especially useful when a viscous absorbing liquid is required. The rate of sampling is necessarily low, 0.25 to 0.5 L/min of air.

S-7 Cold Traps

General Supply Houses (see Table SI-6)

Cold traps (Instrument S-7) are generally component assemblies constructed on an as-needed basis. The cold trap generally consists of a U-shaped glass or copper section which is filled with the adsorbent collection medium. The U-shaped section is immersed in liquid nitrogen or other cold mixture to effect the trapping. The adsorbent used is a function of the contaminant needing collection (e.g., activated carbon is used for organics).

S-8 Plastic Sampling Bags

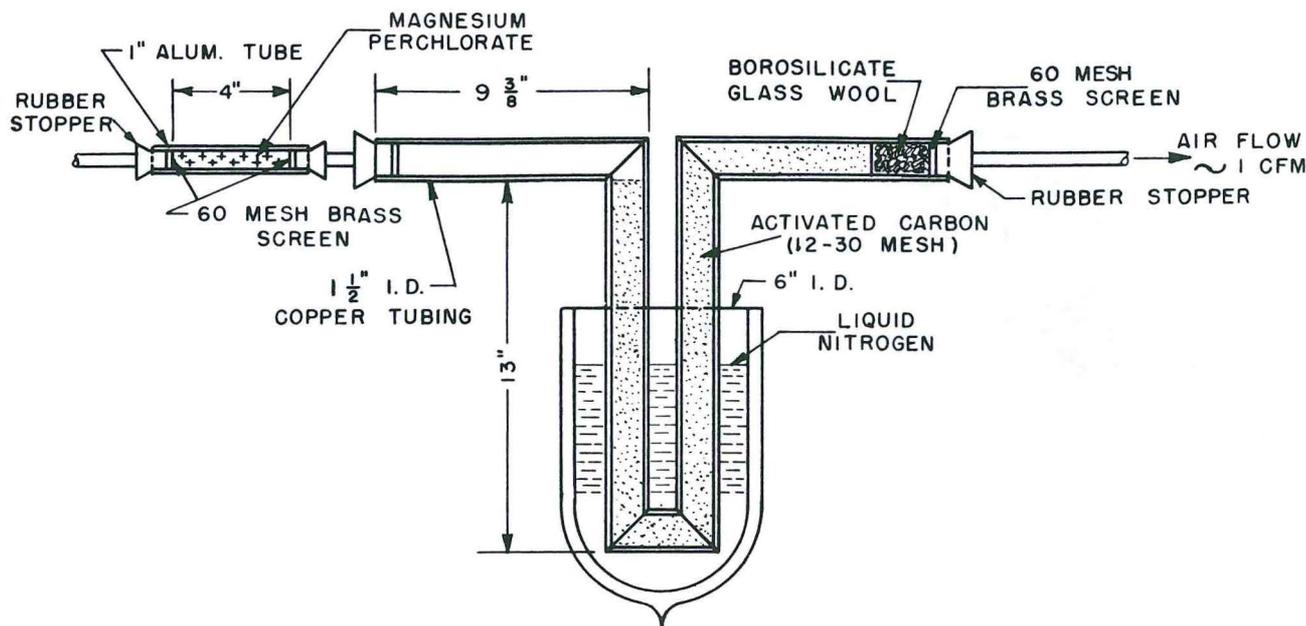
Alltech Associates, Inc.; Anspec Company, Inc.;
Calibrated Instruments, Inc.; Carborundum Plastics, Inc.;
Anatole J. Sipin Company, Inc.

These bags are similar to those used and described under the "Flexible Plastic Containers" section. The difference here is that the bags are used to obtain time-integrated air samples. Air collection systems are available from Calibrated Instruments, Inc., which consist of a six-layer, nonpermeable bag (2-, 5-, 22-, or 44-L capacity), a battery-operated air pump with a belt clip, and an on-off cycle timer.⁽⁸⁶⁾ Sipin provides a similar sampling system which includes a battery-operated air pump (without the timer), a pump belt case, and a protective carrying case for a gas collection bag. This small, day-hike backpack can be worn by a worker for a complete shift if necessary.

S-9 Solid Adsorbents

Barneby Cheney Company;
Columbia Scientific Industries;
Fisher Scientific Company; Pittsburgh;
SKC, Inc.; Westvaco, Inc.; Witco

Several types of charcoal are commercially available. The products used most frequently for air sampling are derived from coconut shells and lignite (Darco and Nuchar). The mesh sizes employed vary considerably. NIOSH recommends 20/40 mesh coconut shell charcoal. Severs and Skory⁽²⁸⁾ found Pittsburgh PCB 12/30 mesh most suitable for sampling vinyl chloride, vinylidene chloride, and methyl chloride. The final choice for a specific application should be made only after per-



INSTRUMENT S-7. Cold trap.

formance and recovery tests have been made.

Sampling tubes for activated charcoal vary in shape and size. NIOSH-recommended tubes measure 7 cm long and 6 mm o.d.^(1,26) The tubes should contain two sections of 20/40 mesh activated charcoal separated by a 2-mm portion of urethane foam. The front end contains 100 mg of charcoal, the backup section, 50 mg. These tubes are commercially available from many chemical suppliers.

Large tubes are also available that contain 600 mg of charcoal, 400 mg in the front section, and 200 mg in the back section; jumbo tubes contain 800 and 200 mg. Other size tubes can be ordered or easily prepared in the laboratory.

Sampling tubes need not always be made of glass. Many in use are constructed of stainless steel. One such unit, described by Severs and Skory,⁽²⁸⁾ measures 5.5 in. \times 0.25 in. o.d. \times 0.028 in. thick and is fitted with Swagelok® caps.

Diffusive Samplers and Monitors

The various commercially available diffusive monitoring systems are listed alphabetically by contaminants in Table SI-1. The tables include manufacturers and brand names. The DuPont colorimetric badge systems are described in Table SI-2. 3M diffusive monitors for ethylene oxide, carbon monoxide, formaldehyde, and mercury are listed in Table SI-3. The types of badges supplied by SKC are found in Table SI-4. Addresses for manufacturers are found at the end of this chapter (Table SI-6). Descriptions of specific systems follow.

Organic Contaminant Samplers

S-10 Pro-Tek™ Badges⁽⁸⁰⁾

E.I. duPont de Nemours & Co., Inc.

The DuPont Pro-Tek G-AA organic vapor air monitoring badge consists of one charcoal strip, two multicavity diffuser elements, and two covers (Instrument S-10). The charcoal strip contains approximately 300 mg of activated charcoal. The badge offers the choice of two sampling rates of approximately 35 and 70 cc/min, depending on whether one or both protective covers are removed. The shortest sampling duration is approximately 0.2 ppm-hours, and the longer duration is limited by saturation of the charcoal strip.

As with charcoal pumped methods, analysis is performed using gas chromatography. Desorption coefficients are determined for all contaminants collected on the charcoal. Published values are available, but they should be determined experimentally for each batch being analyzed.

DuPont's *Sampling Guide* publication lists badge sampling rates for more than 80 compounds. A partial list is found in Table SI-5. Sampling rates with an asterisk were determined experimentally by DuPont. The remaining rates were calculated using published diffusion coefficients or empirical equations modified according to DuPont's experience with its monitor.

The DuPont G-BB Organic Vapor badge is a general purpose device designed to handle most organic vapor requirements. It contains two 300-mg charcoal strips, one in the front section and another in the backup section. It also has two covers and two precision multicavity diffusers. The shortest sampling duration for G-BB



INSTRUMENT S-10. Left to right: Reiszner MiniMonitor; DuPont Pro-Tek™; Abcor GasBadge®; 3M Organic Vapor Monitor #3500.

badges is approximately 0.4 ppm-hours. The longest is again determined by saturation of the charcoal.

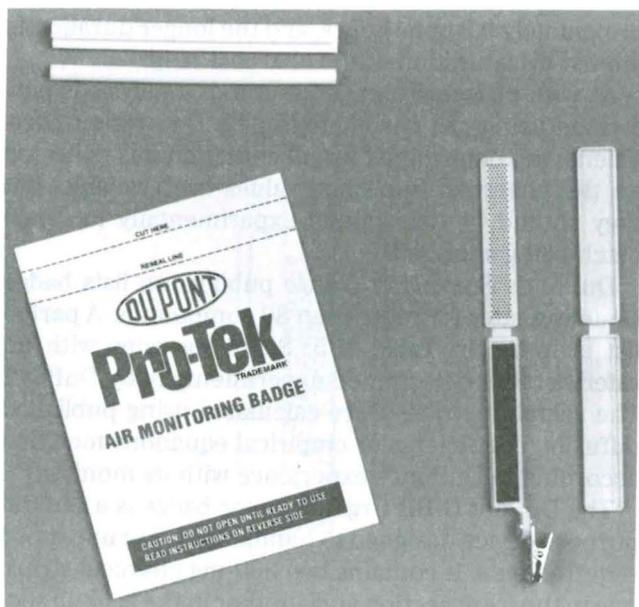
Monitoring instructions and analysis are the same as for the G-AA badge, but the effective weight collected is calculated from:

$$m = m_1 + 2.2 m_2 \quad (12)$$

The factor 2.2 accounts for the increased diffusion path length to the backup section.

S-11 Organic Vapor Monitor #3500 3M Company

This unit⁽⁸⁷⁾ consists of a round nylon body approximately 4.5 cm in diameter, weighing about 12 g



INSTRUMENT S-11. DuPont Pro-Tek™ G-AA organic vapor air monitoring badge.

(Instrument S-11). A charcoal adsorbent pad is located inside the monitor, separated from a diffusion membrane by spacers. Contaminants enter the monitor by molecular diffusion and are adsorbed onto the charcoal. At the end of the sampling period, the diffusion membrane is removed, and a tight fitting cap is snapped into place. The cap contains two ports which are sealed by inserting attached plugs. When the sample is ready for chromatographic analysis, the center port is opened and 1.5 ml of carbon disulfide or other suitable solvent is introduced. The center port is resealed, and the sample is allowed to desorb for 30 minutes.

Desorption efficiency values are known to vary with the amount of material on the charcoal and with type and volume of desorbing solvent used. Therefore, actual desorption efficiencies should always be determined at the time of analysis. The recommended procedure for the 3M #3500 Organic Vapor Monitor is as follows:

The organic compound in the liquid state is introduced through the elutriation port onto a piece of filter paper placed between the elutriation cap and diffusion plate of the monitor. The port is closed and the organic compound is given sufficient time to vaporize and consequently be absorbed by the charcoal sorbent. The filter is removed and analyzed as a separate sample to determine if complete transfer of the organic compound has occurred. Subsequently, the gas chromatographic analysis sample is taken from the center port of the monitor.

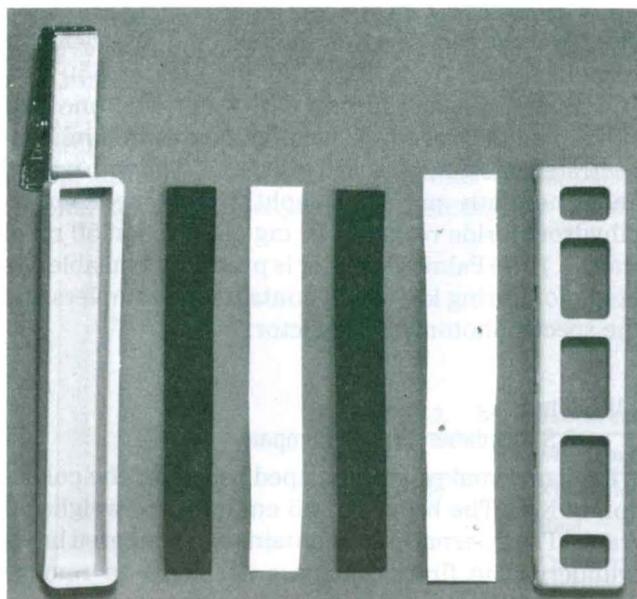
The sampling rate used must be the one supplied by the manufacturer and, if possible, verified by the user. See Table SI-5.

S-12 VaporGard™ Organic Dosimeter⁽⁹⁰⁾ Mine Safety Appliances Company

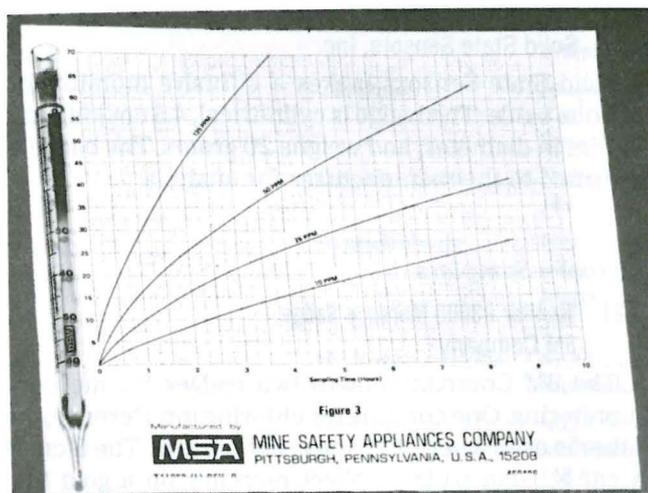
The badge is contained within a plastic strip (Instruments S-12.a and S-12.b) and consists of a wind shield, primary sampling strip, separator, and backup sampling strip. The gaseous contaminant is collected on the primary strip. The backup strip collects the sample after the primary sampling strip becomes saturated. After sampling is done, the charcoal strips are placed in sample envelopes provided and sent to the laboratory for analysis. Both strips are desorbed, analyzed by gas chromatography, and the concentration of gas present is the sum of the mass determined by both analyses. If the backup strip contains more than 10% of the concentration, significant breakthrough has occurred and some of the sample has been lost. VaporGard badges can be used for organic compounds normally collected by charcoal.

S-13 Abcor GasBadge® Organic Vapor Dosimeter⁽⁶¹⁾ National Mine Service Company

This device (see Instrument S-11) collects organic



INSTRUMENT S-12.a. Components of MSA VaporGard™ badge.



INSTRUMENT S-12.b. MSA VaporGard™ inorganic vapor tube with concentration graph.

vapors by diffusion and adsorption on a proprietary activated carbon collection element. The dosimeter measures 5 cm × 6 cm × 1.5 cm, weighs 40 g, and consists of 1) a sliding protective cover; 2) a badge front opening which allows diffusion of gas or vapor into the dosimeter; 3) a draft shield made from non-reactive, porous material; 4) an open grid inside the monitor to define diffusion geometry and minimize internal mixing; 5) a replaceable collection element; and 6) a spring clip.

At the beginning of the exposure period, the sliding protective cover is lowered exposing the front opening and allowing diffusion of gas or vapor into the badge. At

the end of the sampling period, the collection element is removed and analyzed by gas chromatography. An analysis service is provided by the National Mine Service Company. Diffusion coefficients and sampling rates for each organic contaminant are supplied by the manufacturer.

S-14 Reiszner MiniMonitor⁽⁶⁶⁾

Anatole J. Sipin Company, Inc.
Real, Inc.

This device (see Instrument S-11) uses permeation (dimethyl silicone membrane) to control collection of airborne contaminants. Activated charcoal is the collector. The unit measures 5 cm in diameter, is 5 mm thick, and weighs 35 g. The unit is used for the collection of vinyl chloride, but other pollutants can also be measured. The detection limit for vinyl chloride is 0.02 ppm for an eight-hour sample.

S-15 Draeger ORSA 5

National Draeger, Inc.

A compact tube sampler having a diffusion barrier at each end and a 300 mg loading of coconut shell charcoal at the center is available from Draeger as the ORSA 5.⁽⁹²⁾ A special capping and holder arrangement is provided for the tube which is solvent desorbed and analyzed in a similar fashion to the NIOSH charcoal tube method.^(1,26,30) Pannwitz⁽⁹³⁾ has published a comparison of the ORSA 5 sampler with other methods including detector tubes, pumped tubes, and liquid absorption methods. He concluded that there was no essential difference between the results obtained from the active and diffusive methods.

S-16 Automated Thermal Desorber (ATD) Tube

Perkin-Elmer, Ltd.
Perkin-Elmer Corp.

The advantage of a diffusion sampler that could be thermally desorbed was first seen by Brown, Charlton, and Saunders.⁽⁹⁴⁾ This device, which is basically a pumped thermal desorption tube fitted with a diffusive end-cap, is marketed by Perkin-Elmer. Some applications of the sampler and comparisons with pumped methods are given in Brown.⁽⁷⁷⁾ Unlike most other manufactured diffusive samplers, the Perkin-Elmer sampler is supplied empty, and the user packs it with a suitable sorbent for the analyte to be sampled. Some recommendations for suitable sorbents are given in Van den Hoed⁽⁷⁶⁾ or can be obtained from the manufacturer.

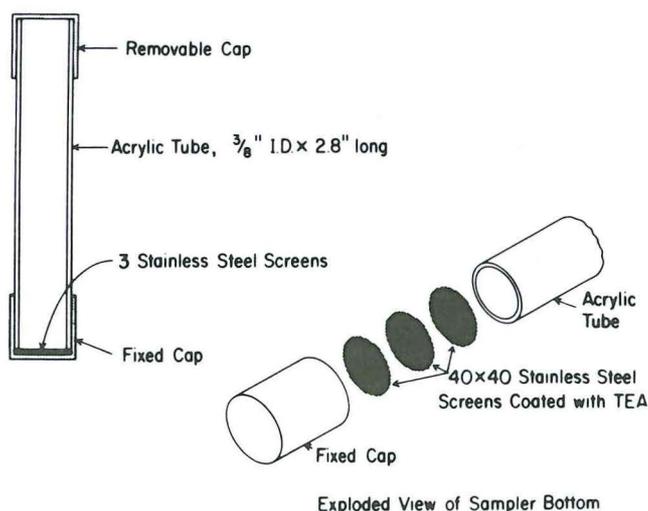
Samplers for Oxides of Nitrogen

S-17 Pro-Tek™ C-30 Colorimetric Badge⁽⁸¹⁾ Pro-Tek Systems, Inc.

This badge is made of an inert polymeric film material which contains a diffuser element (potted hollow fibers), a porous water-repellent tape, and a series of blisters containing color reagents. The badges are packaged in individually sealed pouches weighing 16 grams. Identification stickers, pouch closures, and the recommended analytical method are provided (see Table SI-2).

S-18 Palmes Sampler⁽⁹⁵⁾ Daco Products, Inc. MDA Scientific Company

The Palmes Sampler can be used to sample for both NO_2 and NO_x , determining NO by difference. In both instances, the sampler (Instrument S-18) is constructed of an acrylic tube with a cross-sectional area of 0.71 cm^2 and length of 7.1 cm. Three stainless steel grids coated with triethanolamine for NO_2 reaction/collection (approximately 0.95 mg per screen) in acetone are placed at the bottom of a 1-cm \times 1-cm sleeve-type, low density polyethylene cap. The cap is fixed to one end of the acrylic body. A pen clip and removable cap are added to the other end. The sampler is exposed to NO_2 atmospheres by removing the protective cap. The beginning and ending period of exposure is recorded. The exposure period is terminated by replacing the plastic cap. For collection of NO_x , the described tube additionally has a screen coated with chromic acid (for oxidation of NO to NO_2) and then a second triethanolamine-coated screen for collection of the NO_2 .



INSTRUMENT S-18. Schematic diagram of the Palmes personal NO_2 sampler.

For analysis, 2.1 ml of a combined reagent are added directly into the sampler, mixed, and the detector response read from 10 to 30 minutes at 540 nm. (Combined reagent: to one part water, add one part sulfanilamide reagent [2 g sulfanilamide in 5 ml concentrated phosphoric acid diluted to 100 ml with water] and one-tenth part N-1-naphthylethylene-diamine-dihydrochloride reagent [70 mg dissolved in 50 ml of water].) The Palmes sampler is presently available as a field monitoring kit, which contains the samplers and the spectrophotometric detector.

S-19 Nitrox™ R.S. Landauer, Jr. and Company

Landauer makes a pen-shaped badge for the collection of N_2O . The badge is 14.5 cm long and weighs 46 grams. The internal cavity contains two tempered brass cylinders. The first contains a desiccant to remove water, and the second is a molecular sieve collector for N_2O . The second section is returned to the manufacturer for infrared analysis of the collected N_2O .

S-20 Nitrous Oxide Monitor Solid State Sensors, Inc.

Solid State Sensors makes a diffusive monitor for nitrous oxide. The badge is cylindrical, 4.5 cm long and 3.5 cm in diameter, and weighs 20 grams. The badge is returned to the manufacturer for analysis.

Mercury Samplers

S-21 Monitor #3600 Mercury Badge 3M Company

The 3M Company makes two badges for mercury monitoring. One corrects for chlorine interference, the other is used when chlorine is not present. The 5-cm \times 4-cm \times 1-cm badges collect mercury on a gold film facilitating the formation of a mercury/gold amalgam. The badge is returned to the manufacturer for measurement of the change in electrical conductivity.

S-22 GMD Mercury Badge GMD Systems, Inc.

GMD's Mercury Badge weighs only 14 grams and collects mercury via diffusion and adsorption on Hydrar solid sorbent which is contained in a replaceable capsule element. The sorbent capsule is returned to the laboratory for chemical desorption and analysis by flameless atomic absorption.

S-23 Mercury Badge Solid State Sensors Company

This badge weighs only 9.5 grams and is 5 cm \times 5 cm

× 0.25 cm. Mercury is collected on a gold film. The analytical endpoint (atomic absorption) is provided by the manufacturer.

Other Monitoring Systems

3M diffusive monitors for ethylene oxide, carbon monoxide, formaldehyde, and mercury are listed in

Table SI-3. The types of badges supplied by SKC are found in Table SI-4. Table SI-1 lists various monitoring systems alphabetically along with manufacturers and brand names. Pro-Tek Systems, Inc., sells the Series I and II colorimetric badges for a variety of inorganic gases and vapors. Addresses for manufacturers are found in Table SI-7.

TABLE SI-1. Manufacturers and Brand Names of Diffusion Monitors

| System | Manufacturer | Brand Name |
|------------------|-------------------------------|-----------------------------------|
| Acrylonitrile | Moleculon Research | Poroplastic |
| Alkyl lead | Real | Minimonitor |
| Ammonia | DuPont | Pro-Tek C-10 |
| | SKC | Liquid Sorbent Badge |
| | Moleculon Research MSA | Poroplastic VaporGard |
| Carbon monoxide | MDA Scientific | Monitox |
| | Moleculon Research | Poroplastic |
| | MSA | VaporGard |
| | 3M Willson Safety Products | CO Monitor 3400 Dosimeter Tube |
| Chlorine | Moleculon Research | Poroplastic |
| | Real | Minimonitor |
| | Span, Inc. | Biobadge |
| Ethylene oxide | DuPont | Pro-Tek C-70 |
| | 3M | Monitor 3550 |
| Formaldehyde | DuPont | Pro-Tek C-60 |
| | SKC | Sorbent Badge |
| | Moleculon Research | Poroplastic |
| | 3M | Monitor 3750 |
| Hydrogen cyanide | MDA Scientific | Monitox |
| | Moleculon Research | Poroplastic |
| Hydrogen sulfide | MDA Scientific | Monitox |
| | Moleculon Research | Poroplastic |
| | MSA | VaporGard |
| Mercury | Sipin Environmental | Mercury Badge |
| | SKC | Mercury Vapor Badge |
| | 3M | Monitor 3600 |
| Methanol | SKC | Liquid Sorbent Badge |
| Nitrogen dioxide | Daco Products | Palmes Tube |
| | MDA Scientific | Palmes Tube |
| | Moleculon Research | Poroplastic |
| | MSA | VaporGard |
| | DuPont | Pro-Tek Systems |
| Phosgene | GMD | Phosgene Monitor |
| | SKC | Phosgene Monitor |
| | Moleculon Research | Poroplastic |
| Radon | EDT Instruments | Radon Monitor |
| Sulfur dioxide | DuPont | Pro-Tek C-20 |
| | MSA | VaporGard |
| | Real | Minimonitor |
| Vinyl chloride | Moleculon Research | Poroplastic |
| | Real | Minimonitor |

TABLE SI-2. DuPont Pro-Tek™ Colorimetric Air Monitoring Badge Systems

| System | Absorbing Solution | Reagent Blisters | | | Sampling Range (ppm/hrs) | Shelf Life (Exposed) | Interferences |
|--------------------------|--|---|-------------------------------|---|--------------------------|----------------------|--|
| | | #1 | #2 | #3 | | | |
| Sulfur dioxide (C-20) | 0.004 M potassium tetrachloromercurate with a mercury chloride ratio of 1:16 | Sulfuric acid | 0.11% Formaldehyde | 0.0016% Pararosaniline dye in 2.4 M phosphoric solution | 10-100 | 1 week | Nitrogen dioxide and ozone have slight effect on color intensity |
| Nitrogen dioxide* (C-30) | 0.01 M triethanolamine and 0.01 M butanol | 8% Sulfanilamine, 0.2% N-naphthylene diamine dihydrochloride solution | | | 10-100 | 2 weeks | Strong oxide agents, organic nitrates, sulfur dioxide, and ozone have minor effects on color formation |
| Ammonia (C-10) | 0.3 M boric acid, 0.03 M sodium potassium tartrate solution | 0.022M mercuric iodide, 0.047 M potassium iodide | Pellet of potassium hydroxide | | 50-500 | 2 weeks | Primary organic amines, acetone |
| Formaldehyde (C-50) | 1% aqueous bisulfite | Laboratory analysis by chromotropic acid-sulfuric acid procedure | | | 2-55 | 2 months | n-Butanol, ethanol, phenol, toluene |
| Hydrogen sulfide (C-50) | 5.63×10^{-3} M zinc hydroxide | Laboratory analysis by molybdenum blue method | | | 1.8-164 | 3 months | None by sulfur dioxide, carbon disulfide, ethyl mercaptan, nitrogen dioxide, ammonia, formladehyde |
| Ethyleneoxide (C-70) | 0.05 M sulfuric acid | Laboratory analysis by MBTH method | | | 4-375 | | Water soluble aldehydes |

*See instrument description S-17.

TABLE SI-3. 3M Specific Passive Monitoring Systems

| System | Sampling Range | Interferences | Shelf Life | Analysis |
|--------------------------------|--|---|------------|---------------------------|
| Ethylene oxide | 2 to 600 ppm-hours | None | 1 year | Return to 3M for analysis |
| Carbon monoxide (Monitor 3400) | Endpoint exposure constant 340 ppm-hours | Aromatics and saturated aliphatics | 8 months | Visual analysis |
| Formaldehyde | 0.8 to 72 ppm-hours | Phenol, alcohols, and unsaturated compounds at 10 to 20 times level of formaldehyde | 1 year | Return to 3M for analysis |
| Mercury | Up to 0.20 mg Hg/m ³ | Strong oxidizers such as halogen vapors interfere CO, O ₃ , NO _x , SO ₂ negligible Organic vapors generally do not interfere | 1 year | Return to 3M for analysis |

TABLE SI-4. SKC GA Monitoring Badges

| System | Collection Medium | Analysis | Range |
|-----------------------------|--|--|--|
| Organic Vapor Badges | High activity sorbents* contained in capsules placed in reusable badge housing | Desorption by solvent or heat Analysis by NIOSH methods | Varies with organic compound |
| Liquid Sorbent Badges | | | |
| Ammonia | Ultra high purity water | Colorimetric | 50 ppb to 100 ppm |
| Methanol and other alcohols | Ultra high purity water | Gas chromatographic analysis | 15 to 5000 ppm |
| Formaldehyde | 1% Sodium bisulfite in water | Chromotropic acid | 0.02 to 2 ppm |
| Methyl chloride | Organic solvent | Gas chromatographic analysis | 1.0 to 200 ppm |
| Phosgene | Impregnated badge material | Insert badge in dose determinator | 0.05 to 15 ppm (20-minute exposure) |
| Mercury | Hydrar sorbent | Flameless atomic absorption analysis | As low as 1/25 of TLV (0.05 mg/m ³) 8-hour exposure |

*Anasorb CA for compounds such as ethyl benzene, naphthalene, tetrachloroethylene, trichloroethylene, xylene. Anasorb SR for compounds such as aniline, cresol, dimethyl formamide, ethylamine, morpholine, nitrobenzene, toluidine. Anasorb AK for compounds such as furfuryl alcohol, tetraethyl lead, quinone, ethyl silicate. Anasorb LS (synthetic carbon sorbent). Anasorb EM (sieve type sorbent).

TABLE SI-5. Diffusive Monitoring Sampling Rates (cc/min)

| Compound | DuPont ProTek™ G-AA/G-BB | 3M #3500 Badges Monitor |
|----------------------|-----------------------------|----------------------------|
| Acetone | 38.2 | 35.4 |
| Acrylonitrile | 41.5 | 45.0 |
| Allyl alcohol | 37.2 | 36.2 |
| n-Amyl acetate | 22.2 | 21.6 |
| n-Amyl alcohol | 26.1 | 27.9 |
| Benzene | 35.6* | 33.0 |
| n-Butyl acetate | 24.4 | 23.8 |
| n-Butyl alcohol | 31.4 | 30.5 |
| Carbon tetrachloride | 30.3 | 26.5 |
| Chloroform | 32.9 | 31.6 |
| Cyclohexane | 29.5 | 30.1 |
| Dioxane | 29.7 | 33.6 |
| Heptane | 26.1 | 27.7 |
| Mesityl oxide | 23.3 | 26.9 |
| Methyl ethyl ketone | 33.6* | 31.2 |
| Perchloroethylene | 25.5 | 28.9 |
| Xylene | 27.4 | 25.6 |

*Determined experimentally.
See instrument descriptions S-10 and S-11.

TABLE SI-6. Vendors of Gas and Vapor Collectors

| | | | | | |
|---------------------------------|--|------------------------------------|--|------------------------------|--|
| Plastic Bags | | DPI | Daco Products, Inc. 12 S. Mountain Avenue Montclair, NJ 07042 (201) 744-2453 | WSP | Willson Safety Products P.O. Box 622 Reading, PA 19603 (215) 376-6161 |
| AAI | Alltech Associates, Inc. 2051 Waukegan Road Deerfield, IL 60015 (312) 948-8600 | MSA | Mine Safety Appliances Company 600 Penn Center Blvd. Pittsburgh, PA 15235 (412) 273-5000 | General Supply Houses | |
| ACI | Anspec Company, Inc. 112 Enterprise Drive P.O. Box 7730 Ann Arbor, MI 48107 (313) 665-9666 | SGI | Scientific Glass & Instrument Company P.O. Box 6 Houston, TX 77001 (713) 868-1481 | CPI | Cole Parmer Instrument Co., Inc. 7425 North Oak Park Avenue Chicago, IL 60648 (312) 647-7600 |
| CAL | Calibrated Instruments, Inc. 731 Saw Mill Road Ardsley, NY 10502 (914) 693-9232 | Packaged Sampling Equipment | | EC | Ecology Control 422 Northboro Road Marlboro, MA 01752 (508) 779-5581 |
| CPI | Carborundum Plastics, Inc. 117 State Street Avondale, PA 19311 (215) 268-3101 | AJA | A.J. Abrams Co., Inc. P.O. Box 5171 Westport, CT 06881 (203) 226-4225 | FSC | Fisher Scientific Company 461 Riverside Avenue Medford, MA 02155 (617) 391-6110 |
| MMM | 3M Company 3M Center St. Paul, MN 55101 (612) 733-1110 | CUR | Curtis Matheson 9999 Stuebner Airline Road Houston, TX 77038 (713) 820-9898 | Adsorbents | |
| SUP | Supelco, Inc. Supelco Park Bellefonte, PA 16823 (814) 359-3441 | MDA | MDA Scientific, Inc. 1815 Elmdale Avenue Glenview, IL 60025 (312) 634-2800 | BCC | Barnebey-Cheney Company P.O. Box 2526 Columbus, OH 43216 (614) 258-9501 |
| Bubblers and Gas Washers | | SMI | Sierra-Misco, Inc. 1825 Eastshore Highway Berkeley, CA 94710 (415) 843-1282 | CSI | Columbia Scientific Industries 11950 Jollyville Avenue P.O. Box 203190 Austin, TX 78720 (512) 258-5191 |
| AGI | Ace Glass, Inc. 1430 Northwest Blvd. Vineland, NJ 08360 (609) 692-3333 | ASC | Anatole J. Sipin Company, Inc. 505 Eighth Avenue New York, NY 10018 (212) 695-5706 | PIT | Pittsburgh Division of Calgon Corp. Box 1346 Pittsburgh, PA 15230 (412) 562-8301 |
| BGI | BGI, Incorporated 58 Guinan Street Waltham, MA 02154 (617) 891-9380 | SKC | SKC, Inc. 395 Valley View Road Eighty Four, PA 15330 (412) 941-9701 | WES | Westvaco, Inc. Covington, VA 24426 (703) 962-1121 |
| CGW | Corning Glass Works P.O. Box 5000 Corning, NY 14830 (607) 974-4261 | | | | |

TABLE S-7. Sources of Passive Monitors

| | | | | | |
|-----|---|-----|---|-----|--|
| DRW | Drägerwerk AG Moislinger Alle 53/55 Postfach 1339 D-2400 Lubeck 1, FRG | NDR | National Draeger, Inc. 101 Technology Drive Pittsburgh, PA 15230 (412) 787-8383 | SKC | SKC, Inc. 334 Valley View Road Eighty Four, PA 15330 (412) 941-9701 |
| DUP | E.I. Du Pont de Nemours & Company, Inc. Applied Technology Division Concord Plaza, Clayton Bldg. Wilmington, DE 19898 (215) 444-4035 | NMS | National Mine Service Company Safety Systems & Products U.S. Rt. 22 and 30 West Oakdale, PA 15071 (412) 429-0800 | SSS | Solid State Sensors, Inc. 1974 Ohio Street Lisle, IL 60532 (312) 963-5796 |
| GMD | GMD Systems, Inc. Old Route 519 Hendersonville, PA 15339 412) 746-3600 | PEC | Perkin-Elmer Corp. 761 Main Avenue (MS12) Norwalk, CT 06856 (203) 762-1000 | SPA | Span, Inc. P.O. Box 90279 Houston, TX 77090 (713) 537-2829 |
| LAN | R.S. Landauer, Jr. & Company 2 Science Road Glenwood, IL 60425 (312) 755-7000 | PEL | Perkin-Elmer, Ltd. Post Office Lane Beaconsfield, Bucks HP9 1QA United Kingdom | TEC | Terradex Corp. 460 N. Wiget Lane Walnut Creek, CA 94598 (415) 938-2545 |
| MDA | MDA Scientific Company 405 Barclay Blvd. Lincolnshire, IL 60069 (312) 634-2800 | PRO | Pro-Tek Systems, Inc. 64 Genung Street Middletown, NY 10940 (914) 344-4711 | MMM | 3M Company Occupational Health & Safety Products Division 3M Center, Building 220-3E-D4 St. Paul, MN 55144 (800) 328-1667 |
| MSA | Mine Safety Appliances Company 600 Penn Center Blvd. Pittsburgh, PA 15235 (412) 273-5000 | ASC | Anatole J. Sipin Company, Inc. 505 Eighth Avenue New York, NY 10018 (212) 695-5706 | WSP | Willson Safety Products P.O. Box 622 Reading, PA 19603 (215) 376-6161 |
| MRC | Moleculon Research Corp. Albany Street Cambridge, MA 02142 (617) 577-9900 | | | | |

AIR SAMPLING INSTRUMENTS

for evaluation
of atmospheric
contaminants

7th edition 1989

Susanne V. Hering
Technical Editor



American Conference
of
Governmental Industrial Hygienists
Cincinnati, Ohio

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by
American Conference of Governmental
Industrial Hygienists, Inc.

First Edition 1960
Second Edition 1962
Third Edition 1967
Fourth Edition 1972
Fifth Edition 1978
Sixth Edition 1983
Seventh Edition 1989

Second Printing

Library of Congress Catalog Card Number 83-70265

ISBN: 0-936712-82-1

Published in the United States of America by

American Conference of Governmental Industrial Hygienists, Inc.
6500 Glenway Avenue, Building D-7
Cincinnati, Ohio 45211-4438