

D. GENERAL CONSIDERATIONS FOR SAMPLING AIRBORNE CONTAMINANTS

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1. CHOOSING MEASUREMENT METHODS AND SAMPLING MEDIA

Proper advance planning minimizes sampling and measurement costs and labor and contributes to a smooth, successful survey. Many things must be considered before collecting field samples [1]. The first step is to define sampling objectives. These may include documenting exposures in particular work settings, determining compliance/non-compliance with existing Federal or local standards or recommended exposure limits, or trying to determine the source of a problem. Sampling parameters that should be defined might include type of sample (area vs. personal), contaminant(s) to be sampled, duration of samples, potential interferences and expected contaminant concentrations (or contaminant concentration of interest). Once these parameters are defined, then the proper analytical method and sampling media can be selected. Other general information needed to plan a survey properly include the number of employees, the sampling strategy plan (discussed later), process flow diagram, material safety data sheets on all process materials, the physical states of the substances to be sampled, and potential hazards involved in collecting and shipping the samples.

An accredited analytical laboratory should be used to conduct analysis of collected samples, and it is essential to consult with the analytical laboratory before sampling to ensure that the measurement methods available can meet the defined sampling needs. This step should be an early part of survey planning. The laboratory can also assist in choosing sampling media

that are compatible with the sampling needs and the measurement methods available. The APPLICABILITY section of the individual methods in NMAM can be helpful in choosing which of the available methods is best for a particular situation.

Whether through consultation with the laboratory or through reading the specific measurement method, the sampling media will be specifically identified, e.g., pore size and type of filter, concentration and amount of liquid media required, and specific type and amount of solid sorbent (see Tables 1, 2 and 3 for common types, characteristics and behavior of sampling media). If specific brand name products are called for, no substitutions should be made. Most sampling media are well defined through research and testing; deviations from specifications are undesirable. For example, most organic contaminants are sampled with a dual section tube containing 100 mg front and 50 mg backup sections of 20/40 mesh activated coconut shell charcoal. If larger mesh charcoal or a different type of charcoal were to be used, the sampling capacity and recovery efficiencies for the contaminant of interest might change from that specified in the method.

The physical state of the contaminant(s) being sampled may also be a factor in determining the media required. In the case of polyaromatic hydrocarbons (PAHs), for example, the proper sampler consists of a membrane filter to trap particulate matter and a solid sorbent tube to trap the vapors of certain PAHs so that total collection is assured.

The sampling pump used to collect the sample must also be compatible with the sampling needs and the media used. Specifically, the pump must be capable of maintaining the desired flow rate over the time period needed using the sampling media specified. Some pumps may not be able to handle the large pressure drop of the media. This will be true for fine mesh (smaller than 40 mesh) solid sorbent tubes, small pore size filters or when attempting to take a short-term sample on a sorbent tube of a higher than normal pressure drop at a flow rate of 1 L/min or greater. As a rule of thumb, all high flow pumps (1 to 4 L/min) can handle at least 3 kPa (12 inches of water) pressure drop at 1 L/min for 8 h. Some pumps can handle up to 7.5 kPa (30 inches of water) pressure drop at flows up to 2 or 3 L/min. Most low flow pumps (0.01 to 0.2 L/min) can handle the pressure drops of available sorbent tubes without problems except that the nominal flow rate may decrease for certain models. All pumps should be calibrated with representative sampling media prior to use. It is good practice to check the pump calibration before and after use each day. As a minimum, calibration should be done before and after each survey.

TABLE 1. TYPES AND USES OF SOLID SORBENTS [2]*

Activated charcoal

By far the most commonly used solid sorbent. Very large surface area:wt. ratio. Reactive surface, high adsorptive capacity. This surface reactivity means that activated charcoal is not useful for sampling reactive compounds (e.g., mercaptans, aldehydes) because of poor desorption efficiency. The high capacity, however, makes it the sorbent of choice for those compounds which are stable enough to be collected and recovered in high yield. Breakthrough capacity is a function of type (source) of the charcoal, its particle size and packing configuration in the sorbent bed. Humidity may affect the adsorption as well.

Silica gel

Less reactive than charcoal. Because of its polar nature, it is hygroscopic and shows a decrease in breakthrough capacity for non-water soluble substances with increasing humidity [3].

Porous polymers

Lower surface area and much less reactive surface than charcoal. Adsorptive capacity is, therefore, generally lower, but reactivity is much lower as well.

Ambersorbs™

Properties midway between charcoal and porous polymers.

Coated sorbents

One of the sorbents upon which a layer of a reagent has been deposited. The adsorptive capacity of such systems usually approaches the capacity of the reagent to react with the particular analyte [4].

Molecular sieves

Zeolites and carbon molecular sieves retain adsorbed species according to molecular size. A limiting factor is that the water molecule is of similar size to many small organic compounds and is usually many orders of magnitude higher in concentration than the species of interest. This unfavorable situation may result in the displacement of the analyte by water molecules. Drying tubes may be used during sampling to eliminate the effects of humidity [5].

Thermal desorption

Thermal desorption tubes may contain several different sorbents in order to collect a wide range of different chemicals [6]. These tubes are generally used in situations where unknown chemicals or a wide variety of organics are present, e.g., in indoor environmental air quality investigations. Analysis is often by gas chromatography/mass spectrometry (GC/MS).

***NOTE:** Solid sorbents are used for the collection of vapors only. Aerosols are not collected effectively by most sorbent beds, but may be collected by other components of the sampler (e.g., a prefilter, or the glass wool plugs used to hold the sorbent bed in place).

TABLE 2. TYPES AND USES OF AEROSOL SAMPLERS [6]

Membrane filters

By far the most frequently used filters. This class of filters includes those made from

polyvinyl chloride, Teflon®, silver, and mixed cellulose esters. Filters from this class are used for sampling asbestos, minerals, PAH's, particulates not otherwise regulated, and elements for ICP analysis.

Glass and quartz fiber filters

Quartz filters have replaced glass in many applications. They are used in applications such as sampling for mercaptans and diesel exhaust.

Polycarbonate straight pore filters

Because of their characteristics, these filters are good for the collection of particles to be analyzed by electron microscopy and x-ray fluorescence.

Respirable dust samplers

The 10-mm nylon cyclone and (preferably) conductive cyclones with a 50% cut at 4 µm are used with polyvinyl chloride filters to collect various forms of silica.

Inhalable dust samplers

The Institute of Occupational Medicine's (IOM) sampler is used, in conjunction with a polyvinyl chloride filter, for sampling formaldehyde on dust [7].

TABLE 3. FACTORS AFFECTING THE COLLECTION OF GASES, VAPORS, and AEROSOLS [2, 7]

Temperature

Since all adsorption is exothermic, adsorption is reduced at higher temperatures. Additionally, if there is a reaction between an adsorbed species and the surface, or between two or more adsorbed species (e.g., hydrolysis or polymerization), the rate of such reactions increases at higher temperatures.

Temperature stability of a filter must be considered when sampling hot environments such as stack effluents.

Humidity*

Water vapor is adsorbed by polar sorbents; their breakthrough capacity for the analyte is thereby reduced for most organic compounds. However, for water soluble compounds, the breakthrough capacity is increased, e.g., chlorine and bromine [8] and formaldehyde [3]. This effect varies from substantial for more polar sorbents, such as charcoal and silica gel, to a smaller effect for Amber sorbs™ and porous polymers.

Filter media may also be affected by humidity. Moisture may affect a filter's collection efficiency. Very low humidities (<10% RH) may make some filters (e.g., cellulose ester) develop high charge levels, causing non-uniform deposits and repulsion of particles [9]. Water absorption by some filters (e.g., cellulose ester) can cause difficulty in obtaining tare weights for gravimetric analysis.

Sampling flow rate*/ Face velocity

Breakthrough volume of a solid sorbent bed tends to be smaller at higher sampling flow rates, particularly for coated solid sorbents. For sorbents such as charcoal whose breakthrough capacity for most organic compounds can be significantly reduced by high humidity, lower sampling flow rates may actually result in smaller breakthrough volumes [10]. The collection efficiency of filters will change with face velocity.

Concentration*

As the concentration of contaminant in air increases, breakthrough capacity (mg adsorbed) of a solid sorbent bed increases, but breakthrough volume (L of air sampled) decreases [10].

Particle Characteristics

Filter collection efficiency is a function of pore size [11]. Particles smaller than about 0.2 µm are collected primarily by diffusion, while particles larger than about 0.2 µm are collected primarily by impaction and interception. Most sampling filters are highly efficient (>95%) for all particle sizes, with the minimum efficiency in the 0.2 µm size range. Polycarbonate straight pore filters exhibit poor collection by diffusion, so particles smaller than the pore size are not collected efficiently.

Filter considerations

The pressure drop of a filter can limit the sampling time, because of the load on the personal sampling pump. In addition, pressure drop increases with dust loading on the

filter. Fine particles (#0.5 μm) will increase the pressure drop much faster than coarse particles (10 μm). Heavy loading (about 1 mg) may result in poor adhesion of collected particles to the filter surface.

***NOTE:** It is important to distinguish between equilibrium (saturation) adsorptive capacity and kinetic (breakthrough) adsorptive capacity of the solid sorbent. Breakthrough capacity is the important characteristic in actual sampling situations; it may be affected significantly by sampling flow rate and relative humidity of the air being sampled and may be significantly less than saturation capacity, which is not dependent on sampling flow rate or relative humidity.

2. FIGURING SAMPLING PARAMETERS

Once the sampling media and measurement method are chosen, then the specific sampling parameters need to be determined [12]. For most methods, this will not pose a problem as the flow rate recommended in the method can be used for the desired sampling period, e.g., 1 to 3 L/min for 8 h for most aerosols or 10 to 200 mL/min for 8 h for most sorbent tube samples. Generally, the parameters which must be considered are flow rate, total sample volume, sampling time (tied into the two previous parameters), and limit of quantitation (LOQ) (see Glossary of Abbreviations, Definitions and Symbols). Some of these variables will be fixed by sampling needs, e.g., sampling time or by the measurement method of choice (LOQ or maximum sampling volumes). The choice of these variables can best be explained through the use of the following examples.

Examples:

a. Sampling for Gases and Vapors Using Solid Sorbents

Given parameters:	Method 1501 for Styrene
Recommended Sample Volume:	5 L
Useful Range of the Method:	85 to 2560 mg/m^3 (20 to 600 ppm)
OSHA PEL:	850 mg/m^3 (200 ppm) - Ceiling 425 mg/m^3 (100 ppm) - TWA
Recommended Flow Rate:	0.2 L/min
Breakthrough Time:	111 min @ 0.2 L/min and 1710 mg/m^3
Breakthrough Capacity	38 mg

Suppose it is desired to determine both ceiling and TWA exposures of workers exposed to styrene and the concentrations are unknown.

Ceiling Determination: If sampling were done at 0.2 L/min for 30 min and a total sample volume of 6 L collected which is above the 5 L recommended sample volume, would this a problem? Probably not. For instance, in the breakthrough test, a concentration of 2 times the OSHA Ceiling Standard (1710 mg/m^3) was sampled at 0.2 L/min for 111 min (22.2 L) before breakthrough occurred, collecting a total weight of 38 mg of styrene. Of course, this test was conducted in a dry environment with only styrene present. A safety factor of 50% should be allowed to account for humidity effects. Thus, if sampling is done for about 55 min at 0.2 L/min, levels of styrene up to 400 ppm could still be collected without sample breakthrough.

Also to be considered are the other organics present. If a concentration of 200 ppm acetone exists in this environment, then an additional safety factor should be added. An arbitrary 50%

reduction in total sampling time or 28 min at 0.2 L/min might be done. This is very close to the original sampling time of 30 min. With the safety factors built in, collecting a 6-L sample should not be a problem. Alternately, the flow could be reduced to 0.1 L/min and be well within the 5-L total volume.

TWA Determination: In this same situation, the goal is to collect 8-h samples for comparison to the 100 ppm TWA. If sampling were done at 0.05 L/min, then the total sample volume would be 22.5 L, substantially above the 5-L recommended sample volume. If the flow was dropped to 0.02 L/min, then the sample volume would be 9 L. This sample volume might be acceptable if the styrene concentrations are around 100 ppm and no other competing organics are present, e.g., acetone. However, the safer approach would be to collect two consecutive samples at 0.02 L/min for 4 h (total sample volume of 4.8 L each).

b. Pushing a Method to the Limit, Limit of Quantitation

Given Parameters:	Method 1009 for Vinyl Bromide (VB)
Recommended Sample Volume:	<10 L @ 0.20 L/min or less
Working Range:	0.3 to 33 ppm (1.3 to 145 mg/m ³) for a 6-L air sample; this equals 8 to 355 µg VB per tube
Limit of Detection:	3 Fg VB per tube

In this particular example, let us say that the object is to estimate exposure to vinyl bromide down to 0.1 ppm (0.44 mg/m³), which is below the working range. In order to collect 8 µg of vinyl bromide (the limit of quantitation) at this concentration, 20 L of air will have to be collected. This volume is substantially above the maximum recommended sample volume of 10 L. Since the recommended sample volume is generally a conservative value used to protect against breakthrough under worst case conditions (i.e., high humidity and high concentrations), considerable leeway exists for the size of the air sample. In this example, the 20-L air samples should be taken at 0.2 L/min or slower, and the possibility of breakthrough should be monitored by observing the relative amounts of analyte on the backup sections of the samples.

The best approach is to consult with the analytical laboratory and then to take a sufficient number of samples to determine the useful limits of the sampler in the particular application. The presence of high relative humidity and other organic solvents will severely reduce the number of active sites available on the sorbent for collection of the contaminant of interest. In pushing a method to the limit, it is often necessary to sample beyond the breakthrough volume, while observing recommended maximum sampling flow rate, in order to obtain the sensitivity to determine the concentration of interest. If this is done, then the risk must be accepted that the method may not work outside the limits tested.

c. Sampling for Dusts Using a Membrane Filter

Given parameters:	Method 7401 for Alkaline Dusts
Recommended Sample Volume:	70 L @ 2 mg/m ³ ; 1000 L max.
Useful Range of the Method:	0.76 to 3.9 mg/m ³

OSHA PEL:	2 mg/m ³ (NaOH)
Recommended Flow Rate:	1 to 4 L/min

Suppose it is desired to determine both an exposure taking place during a specific 20 minute operation as well as a TWA exposure of workers exposed to sodium hydroxide and the concentrations are unknown.

20 Minute Process Sample: This sample would meet the method conditions by sampling for the 20 minutes at 4 L/min since this would collect 80 L. Sampling at 1, 2, or 3 L/min for 20 minutes would probably not allow for the collection of sufficient sample required for analysis.

TWA Determination: In this situation, it is necessary to collect an 8-h sample to compare with the 2 mg/m³ OSHA PEL. Since an 8-h TWA sample covers 480 minutes, sampling can no longer be done at 4 L/min since this would collect 1920 L, almost twice the upper recommended sample volume. Sampling at 1 L/min would collect a 480 L sample, and sampling at 2 L/min would collect a 960 L sample, both acceptable per the conditions of the method.

3. BULK SAMPLES

The addition of bulk samples can often make the difference between a successful or unsuccessful sampling effort. This is especially true where there is mixed solvent exposure or unknown dust exposure and for determining silica content of dusts. The primary purpose of bulk samples is to provide the analytical laboratory with a large enough sample for qualitative and sometimes quantitative analysis. The two major types of bulk samples are bulk air and mass bulk (liquid or solid) samples.

a. Bulk Air Samples

Generally, a bulk air sample is defined as a large volume area sample collected for the purpose of qualitative analysis. A good example is multiple solvent exposure where the exact identity of the airborne solvents is unknown, e.g., painting operations. For most organic solvents, a bulk air sample consists of a charcoal tube (or whatever sorbent is called for) collected at 1 L/min for an hour or more. Although the sample is likely to exhibit breakthrough, this does not matter since one is primarily interested in what substances are present rather than their exact concentrations (the latter aim is accomplished through the separate collection of proper samples). Any questions concerning how or whether or not a bulk air sample is needed should be addressed to the analytical laboratory prior to sampling. In the case of silica, either a bulk air or solid bulk sample (e.g., a rafter sample) or both are suggested so that enough material will be available to determine free silica content.

b. Bulk Liquids and Solids

Collection of bulk materials may be needed to establish the substances present in the workplace and, in some cases, to establish the relative levels of certain substances present in the raw material. A good example of the latter is the case of mixed solvent exposure when determining if a certain contaminant of interest is present, e.g., benzene. In some cases, a list of 30 solvents may be present (from Material Safety Data Sheets), but it is not certain

which ones are present or in what proportions. This example is also true for dusts, as was discussed previously for silica, or for metals which may exist in trace quantities.

In choosing bulk samples, the end goal must be considered: qualitative and/or quantitative analysis. In the case of a painting operation, it is preferred to have the bulk samples separated by contaminants of interest, i.e., solvent fraction separate from the pigment fraction. This allows the laboratory to analyze the different portions of the paints without having to go through a lengthy separation process. In general, the cleaner the bulk, the easier it will be for the laboratory to conduct the analysis. In many cases, the industrial hygienist is interested in a "dirty" bulk. Any information that can be given to the laboratory on what may or may not be present will help speed up the analysis. Advance consultation with the laboratory is desirable.

In choosing bulk dust samples, the sample should be representative of the airborne dust to which the workers are being exposed. Usually this is a settled dust sample collected from rafters or near the workers' job site. In other cases, a process dust sample is chosen to determine the composition of the material before it is airborne. In cases where the choice is not clear, do not follow the adage that "more is better." Bulk samples should be limited in number to optimize the laboratory's time. A good approach, when in doubt as to what bulks are needed, is to collect several but to allow the laboratory to analyze only those needed to answer questions as they arise.

When shipping bulks, care must be taken to preserve the integrity of the samples and to follow established Department of Transportation (DOT) shipping regulations. Only 5 to 10 mL of the liquid or solid is needed, so keep bulk sample sizes small. In general, leak-proof glass containers are best since they will not react with most chemicals; however, polyethylene containers can be used in the majority of cases. A convenient container is a 20-mL scintillation vial with PTFE-lined cap. Specific chemicals for which polyethylene containers should not be used include aromatic compounds, chlorinated hydrocarbons and strong acids. The lids of the containers should be sealed with shrink bands or tape for further assurance against leakage. These containers should be labeled as required by DOT under their regulations, 49CFR Part 171-177. For most materials classified as "Flammable" or "Poisonous," amounts up to 1 quart can be shipped by any carrier. Most bulk dusts are not covered by DOT regulations. Specific restrictions and labeling requirements should be checked prior to shipping any samples.

In the case of volatile bulk samples (and some air samples), consideration should be given to shipping the samples on dry ice or with bagged refrigerant (e.g., "blue ice"). Do not ship volatiles together with air samples. Again, check with the carrier you plan to use as there

may be restrictions on the amount of dry ice they will accept in a package (usually 5 pounds or less is acceptable). Specific labels are usually required when dry ice is used.

4. BLANKS

Certain numbers of blanks are required by the analytical laboratory for each set of samples to be analyzed. The specific method being used should be consulted concerning the number and type of blanks required. There are two types of sampler blanks: field and media blanks.

Field blanks are clean samplers taken to the sampling site, handled in every way as the air samples, except that no air is drawn through them. Media blanks are simply unopened, new samplers which are sent with the samples (these blanks are not usually taken to the field). It is also recommended that additional blind field blanks be sent along with the field samples, labeled as field samples, as a further check on the analysis. Blanks are good insurance to deal with contamination, but the best approach is to avoid sample contamination by being careful. The recommended practice for the number of field blanks is two field blanks for each 10 samples with a maximum of 10 field blanks for each sample set. Media blanks should also be included. These unexposed, unopened samplers will give an estimate of media background. The laboratory should analyze at least six (6) media blanks from the same lot as the field samples. This number should be increased for media which are coated or impregnated with reagent. Again, consult the specific method for the number and type of blanks as these numbers will vary.

Another frequently-used practice is to include blind spiked samples as a quality control check of the analytical laboratory. See the following chapter for a detailed discussion of spiking procedures.

5. DIRECT-READING METHODS

The variety of types of direct-reading methods available is large and expanding, including detector tubes (both short- and long-term), aerosol monitors, integrating passive monitors for certain gases and portable instrumentation for gas chromatography or infrared spectroscopy [13]. Many direct-reading instruments now used for personal or area measurements have evolved from laboratory or process control instruments [14].

Some of the considerations (i.e., specificity and sensitivity) for the use of direct-reading methods for quantitative determinations are similar to those already given for classical filter or sorbent methods. In many cases, direct-reading instruments, which are physically small and portable, qualify as personal sampling devices.* These offer the additional advantages over classical methods by reducing labor and analytical costs and may be the methods of choice when instantaneous results are important, even at the expense of some degree of sensitivity or specificity. In general, manufacturers' instructions should be followed in the calibration and use of these devices. Because of the severe conditions to which direct-reading instruments may be subjected, performance checks and preventive maintenance on a periodic basis or before each use are very important. Many direct-reading instruments are powered by Ni-Cd batteries which can fail to provide a full charge over the full sampling period unless frequently or fully discharged and recharged several

times just prior to use. An additional responsibility, that of field calibration of the direct-reading instrument, falls on the field sampling personnel.

***NOTE:** A portable instrument is defined as weighing less than 4.5 kg (10 lbs.) and powered by self-contained batteries [15]. For personal monitoring, the instrument configuration should be such that the breathing zone can be monitored. Alarms, both audible and visual, and hard-copy documentation are desirable.

6. SAMPLING STRATEGY

To obtain the maximum amount of information during the course of a survey with a minimum number of samples, a statistical sampling strategy should be developed before conducting any survey [16]. Several pieces of information must be known in advance to plan a sampling strategy, including the size of the workforce to be sampled, the accuracy of the sampling and measurement method to be used and the confidence one wishes to have in predicting the exposure of the workforce.

For example, to determine with 90% confidence that at least one worker from a workplace subgroup will be in the top 10% of the exposures occurring in the group, the number of employees to sample would be chosen from Table 4. Other tables are given in the publication for confidence limits of 95% and for the top 20% of exposures. Again, judicious use of sampling statistics will optimize the number of samples needed.

Table 4. Minimum sample size (n) for including (@ 90% confidence level) at least one high risk employee* [16]

Size of Employee Group (N):																			
1	2	3	4	5	6	7	8	9	10	11-12	13-14	15-17	18-20	21-24	25-29	30-37	39-49	50	4

Minimum Number of Measured Employees (n):																			
1	2	3	4	5	6	7	7	8	9	10	11	12	13	14	15	16	17	18	22

*Exposure in highest 10% of N.

7. SAMPLING AND CALIBRATION TECHNIQUES

The following are suggested general techniques for active sampling using some of the more common samplers. These instructions elaborate on those given in NMAM methods. Consult individual methods for details of sample size.

a. Calibration of Personal Sampling Pumps

The accuracy of determining the concentration of a toxic substance in air is no greater than the accuracy with which the air volume is measured. Therefore, accurate calibration of the airflow rate through the sampling train is necessary. The frequency of calibration depends on the use, care and handling to which the pump is subjected. In addition, pumps must be recalibrated after each repair and if they have been abused. Ordinarily, pumps should be calibrated in the laboratory and the field, both before field use and after each field survey.

The choice of a reference instrument will depend on where the calibration is to be performed. For laboratory use, primary standards, such as a spirometer or soap-bubble meter, are recommended [17]. Several electronic soap-bubble calibrators and one dry-cell calibrator are commercially available as primary calibrators. Other instruments, such as a wet-test,

mass-flow or a dry-gas meter, may be used. The following instructions are for the soap-bubble meter. If another instrument is used, equivalent procedures should be followed.

- (1) Set up the apparatus as shown in Figure 1.
- (2) Make certain that the rechargeable batteries will power the pump for the entire sampling interval by one of the following methods: 1) run the pump for that length of time, checking for satisfactory operations; 2) test the battery independently of the pump using a current capacity tester [17]. Fully recharge the batteries.
- (3) Turn the pump on and moisten the inner surface of the soap-bubble meter with the soap solution. Draw bubbles upward until they travel the entire length of the buret without breaking.
- (4) Adjust the pump to the desired nominal flow rate. Check the water manometer. The pressure drop across the sampler should not exceed 2.5 cm Hg (13 inches) of water.
- (5) Start a soap bubble in the buret and measure the time, with a stopwatch, that it takes to traverse two calibration marks. For a 1000-mL buret, a convenient calibration volume is 500 mL. Repeat the determination at least twice more. Average the results and calculate the flow rate by dividing the calibration volume by the average time.
- (6) Record the following data:
 - a. volume measured
 - b. elapsed time
 - c. pressure drop
 - d. air temperature
 - e. atmospheric pressure
 - f. serial number of the pump
 - g. pump model
 - h. date and name of operator
- (7) If the sampling pump used for sample collection uses a variable area flow meter (rotameter) for flow rate indication, the calibrated flow rate must be adjusted for the actual air pressure and temperature during sampling [18]. The expression for this correction is as follows.

NOTE: This correction is **not** used for non-rotameter sampling pumps.

$$V \text{ (Corrected volume, L)} = Q t (P_c T_s / P_s T_c)^{0.5}$$

where: Q = indicated flow rate (L/min)
 t = sampling time (min)
 P_c = pressure during calibration of sampling pump

(kPa or other pressure units)
 P_s = pressure of air sampled (same units as P_c)
 T_c = temperature during calibration of sampling pump (EK)
 T_s = temperature of air sampled (EK).

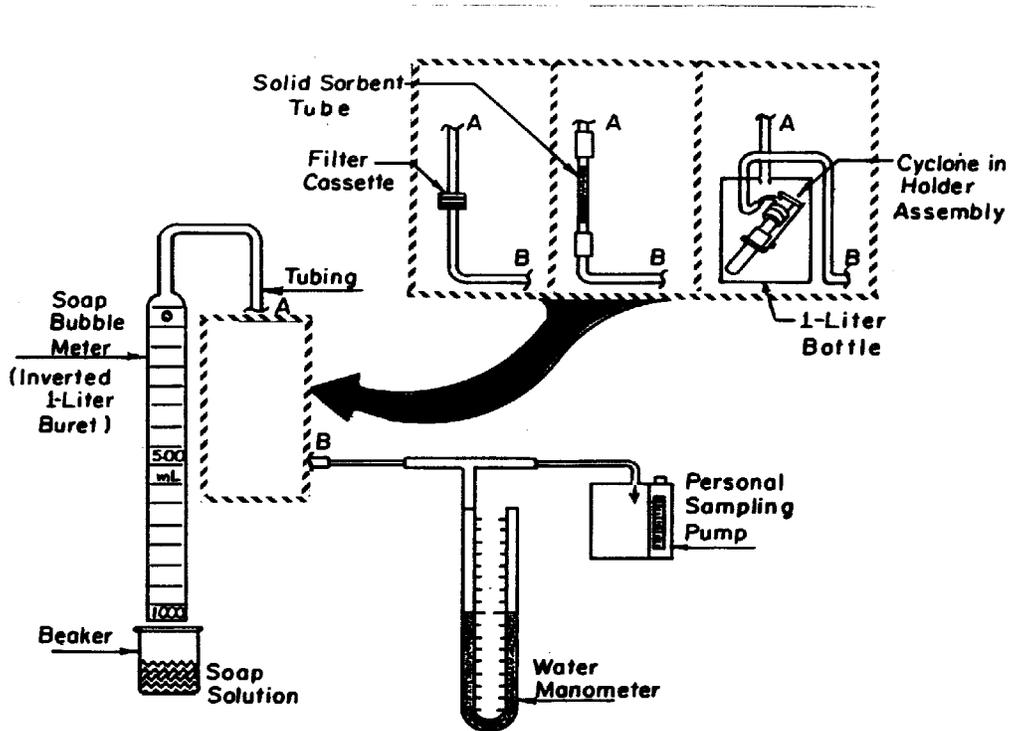


Figure 1. Calibration Apparatus.

b. Sampling Instructions for Solid Sorbent Tube Sampler

Use these instructions for active personal sampling (i.e., pumped sample airflow) for substances which are retained on solid sorbents such as activated charcoal, silica gel, porous polymers, etc.

- (1) Calibrate each personal sampling pump at the desired flow rate with a representative solid sorbent tube in line (alternatively, use a flow restrictor to provide a pressure drop equal to that of the average solid sorbent tube). Use a bubble meter or equivalent flow measuring device.
- (2) Break the ends of the solid sorbent tube immediately before sampling to provide an opening at least one-half of the internal diameter at each end.

- (3) Connect the solid sorbent tube to a calibrated personal sampling pump with flexible tubing with the smaller sorbent section (backup section) nearer to the pump. Do not pass the air being sampled through any hose or tubing before entering the solid sorbent tube. Position the solid sorbent tube vertically during sampling to avoid channeling and premature breakthrough.
- (4) Prepare the field blanks at about the same time as sampling is begun. These field blanks should consist of unused solid sorbent tubes from the same lot used for sample collection. Handle and ship the field blanks exactly as the samples (e.g., break the ends and seal with plastic caps) but do not draw air through the field blanks. Two field blanks are required for each 10 samples with a maximum of 10 field blanks per sample set.
- (5) Take the sample at an accurately known flow rate as specified in the method for the substance and for the specified air volume. Typical flow rates are in the range 0.01 to 0.2 L/min. Check the pump during sampling to determine that the flow rate has not changed. If sampling problems preclude the accurate measurement of air volume, discard the sample. Take two to four replicate samples for quality control for each set of field samples.
- (6) Record pertinent sampling data including location of sample, times of beginning and end of sampling, initial and final air temperatures, relative humidity and atmospheric pressure or elevation above sea level.
- (7) Seal the ends of the tube immediately after sampling with plastic caps. Label each sample and blank clearly with waterproof identification.
- (8) Pack the tubes tightly with adequate padding to minimize breakage for shipment to the laboratory. In addition to the sample tubes and field blanks, ship at least six unopened tubes to be used as media blanks so that desorption efficiency studies can be performed on the same lot of sorbent used for sampling.
- (9) Ship bulk samples in a separate package from the air samples to avoid contamination of the samples. Suitable containers for bulk samples are glass with a polytetrafluoroethylene (PTFE)-lined cap, e.g., 20-mL glass scintillation vials.

c. Sampling Instructions for Filter Sampler

Use these instructions for personal sampling of total (respirable and non-respirable) aerosols. Methods requiring these instructions specify FILTER as the sampling method. These instructions are not intended for respirable aerosol sampling.

- (1) Calibrate the personal sampling pump with a representative filter in line using a bubble meter or equivalent flow measuring device.
- (2) Assemble the filter in the two-piece cassette filter holder. Support the filter by a cellulose backup pad or stainless steel screen. Close the cassette using a press

[19] or other technique that ensures cassette parts mate evenly and positively to prevent leakage. Seal the filter holder with plastic tape or a shrinkable cellulose band to reduce contamination during filter removal.

- (3) Remove the filter holder plugs and attach the filter holder to the personal sampling pump with a piece of flexible tubing. Clip the filter holder to the worker's lapel. Air being sampled should not be passed through any hose or tubing before entering the filter holder.
- (4) Prepare the field blanks at about the same time as sampling is begun. These field blanks should consist of unused filters and filter holders from the same lot used for sample collection. Handle and ship the field blanks exactly as the samples, but do not draw air through the field blanks. Two field blanks are required for each 10 samples with a maximum of 10 field blanks per sample set.
- (5) Sample at a flow rate of 1 to 3 L/min until the recommended sample volume is reached. Set the flow rate as accurately as possible (e.g., within $\pm 5\%$) using the personal sampling pump manufacturer's directions. Take two to four replicate samples for quality control for each set of field samples.
- (6) Observe the sampler frequently and terminate sampling at the first evidence of excessive filter loading or change in personal sampling pump flow rate. (It is possible for a filter to become plugged by heavy particulate loading or by the presence of oil mists or other liquids in the air.)
- (7) Disconnect the filter holder after sampling. Cap the inlet and outlet of the filter holder with plugs. Label the sample. Record pertinent sampling data including times of beginning and end of sampling, initial and final air temperatures, relative humidity and atmospheric pressure or elevation above sea level. Record the type of personal sampling pump used and location of sampler.
- (8) Ship the samples to the laboratory as soon as possible in a suitable container designed to prevent damage in transit. Ship bulk material to the laboratory in a glass container with a PTFE-lined cap. Never store, transport or mail the bulk sample in the same container as the samples or field blanks. In addition to the samples and field blanks, ship five unopened samplers from the same lot for use as media blanks.

d. **Sampling Instructions for Filter + Cyclone Sampler**

Use these instructions for personal sampling of respirable aerosols (ACGIH definition [20]). Methods requiring these instructions specify CYCLONE + FILTER as the sampling method.

- (1) Calibrate the pump to the rate specified by the cyclone (1.7 L/min for the 10-mm nylon cyclone, 2.2 L/min for the Higgins-Dewell cyclone [21], or 2.5 L/min for the aluminum cyclone), with a representative cyclone sampler in line using a bubble meter or a secondary flow measuring device which has been calibrated against a

bubble meter. The calibration of the personal sampling pump should be done close to the same altitude where the sample will be taken.

- (2) Assemble the pre-weighed filter in the two-piece cassette filter holder. Support the filter with a cellulose backup pad or stainless steel screen. Close firmly to prevent sample leakage around the filter. Seal the filter holder with plastic tape or a shrinkable cellulose band.
- (3) Remove the cyclone's grit cap and vortex finder before use and inspect the cyclone interior. If the inside is visibly scored, discard this cyclone since the dust separation characteristics of the cyclone might be altered. Clean the interior of the cyclone to prevent reentrainment of large particles.
- (4) Assemble the two-piece filter holder, coupler, cyclone and sampling head. The sampling head rigidly holds together the cyclone and filter holder. Check and adjust the alignment of the filter holder and cyclone in the sampling head to prevent leakage. Connect the outlet of the sampling head to the personal sampling pump by a 1-m piece of 6-mm ID flexible tubing.
- (5) Clip the cyclone assembly to the worker's lapel and the personal sampling pump to the belt. Ensure that the cyclone hangs vertically. Explain to the worker why the cyclone must not be inverted.
- (6) Prepare the field blanks at about the same time as sampling is begun. These field blanks should consist of unused filters and filter holders from the same lot used for sample collection. Handle and ship the field blanks exactly as the samples, but do not draw air through the field blanks. Two field blanks are required for each 10 samples with a maximum of 10 field blanks per sample set.
- (7) Turn on the pump and begin sample collection. If necessary, reset the flow rate to the pre-calibrated value, using the manufacturer's adjustment procedures. Since it is possible for a filter to become plugged by heavy particulate loading or by the presence of oil mists or other liquids in the air, observe the filter and personal sampling pump frequently to keep the flow rate within $\pm 5\%$ of the target flow rate. The sampling should be terminated at the first evidence of a problem.
- (8) Disconnect the filter after sampling. Cap the inlet and outlet of the filter holder with plugs. Label the sample. Record pertinent sampling data including times of beginning and end of sampling, initial and final air temperatures and atmospheric pressure or elevation above sea level. Record the type of personal sampling pump, filter, cyclone used and the location of the sampler.
- (9) Ship the samples and field blanks to the laboratory in a suitable container designed to prevent damage in transit. Ship bulk samples in a separate package.
- (10) Take two to four replicate samples for every set of field samples to assure quality of

the sampling procedures. The set of replicate samples should be exposed to the same dust environment, either in a laboratory dust chamber or in the field. The quality control samples must be taken with the same equipment, procedures and personnel used in the routine field samples. The relative standard deviation, s_r , calculated from these replicates should be recorded on control charts and action taken when the precision is out of control.

e. Jarless Method of Calibration of Cyclone Assemblies [23]

This procedure may be used in the field to calibrate an air sampling pump and a cyclone assembly without using the one-liter "calibration jar."

- (1) Connect the pump to a pressure gauge or water manometer, a light load equal to 2 to 5" H₂O, and an electronic bubble meter or standard bubble tube.
- (2) Adjust the pump to 1.7 L/min as indicated on the bubble meter/tube, under the light load conditions (2 to 5" H₂O) as indicated on the pressure gauge or manometer.
- (3) Increase the load until the pressure gauge or water manometer indicates between 25 and 35" H₂O. Check the flow rate of the pump again. The flow rate should remain at 1.7 L/min \pm 5%.
- (4) Replace the pressure gauge or water manometer and the electronic bubble meter or standard bubble tube with the cyclone having a clean filter installed. If the loading caused by the cyclone assembly is between 2 and 5" H₂O, the calibration is complete and the pump and cyclone are ready for sampling.

8. REFERENCES:

- [1] First MW [1995]. Air Sampling and Analysis of Air Contaminants: An Overview. In: Cohen BS, Hering SV, Eds. Air Sampling Instruments, 8th ed. Cincinnati, OH: ACGIH, pp. 1-18.
- [2] Melcher RG [1987]. Laboratory and Field Validation of Solid Sorbent Samplers. In: Taylor JK, Ed. Sampling and Calibration for Atmospheric Measurements, ASTM STP 957, Philadelphia PA: American Society for Testing and Materials, pp. 149-165.
- [3] Iraneta PC, Collamati RA, Costello DN, Crowley RJ, Fernandez RL, Hopkins MR, Knowles CE, Martin DM [in press]. A validation study for a new active sampler for the analysis of formaldehyde in workplace and indoor air. Am Ind Hyg Assoc J.
- [4] Kennedy ER [1988]. Sampling Reactive Materials. In: Advances in Air Sampling, ACGIH. Chelsea, MI: Lewis Publishers, pp.175-188.
- [5] Langhorst M [1983]. A Hollow Fiber Device for Separating Water Vapor from Organic Vapors. Am Ind Hyg Assoc J 44 :592.
- [6] Hodgson AT, Binenboym J, German JR [1988]. A Multisorbent Sampler for Volatile Organic Compounds in Indoor Air. In: Advances in Air Sampling, ACGIH. Chelsea, MI: Lewis Publishers, pp. 143-157.
- [7] NIOSH [1994]. Formaldehyde on Dust: Method 5700. In: Eller PM, Cassinelli ME,

- eds. NIOSH Manual of Analytical Methods (NMAM), 4th ed. Cincinnati, OH: National Institute for Occupational Safety and Health, DHHS (NIOSH) Publication No. 94-113.
- [8] Lippmann M [1995]. Filters and Filter Holders. In: Cohen BS, Hering SV, Eds. Air Sampling Instruments, 8th ed. Cincinnati, OH: ACGIH, pp. 247 - 278.
- [9] Cassinelli ME [1991]. Development of a solid sorbent monitoring method for chlorine and bromine with determination by ion chromatography. *Appl Occup Environ Hyg* 6:215-226.
- [10] Chen CC, Baron PA [1996]. Aspiration Efficiency and Wall Deposition in the Fiber Sampling Cassette," *Am Ind Hyg Assoc J*, 52: 142-152.
- [11] Foerst D [1979]. A Sampling and Analytical Method for Vinylidene Chloride in Air. *Am Ind Hyg Assoc J*, 40:, 888-893.
- [12] Lee KW, Ramamurthi M [1993]. Filter Collection. In: Willeke KW, Baron PA, Eds. Aerosol Measurement. New York: Van Nostrand Reinhold.
- [13] Eller PM [1986]. Operational Limits of Air Analysis Methods. *Appl Ind Hyg* 1(2): 91-94.
- [14] Wuebkenberg ML, McCammon CS [1995]. Direct-Reading Gas and Vapor Instruments. In: Cohen BS, Hering SV, Eds. Air Sampling Instruments, 8th ed. Cincinnati, OH: ACGIH, pp 439-510.
- [15] Enviro Control, Inc. [1981]. Assessment of Engineering Control Monitoring Equipment, Final Report, NIOSH Contract 210-79-0011, available from NTIS, Springfield, VA 22161 as Order Nos. PB83-152629 (Vol. 1) and PB83-152637 (Vol. 2).
- [16] Wuebkenberg ML, Woodfin WJ [1983]. NIOSH-developed systems for monitoring equipment evaluations. In: Proceedings of third annual national symposium on recent advances in the measurement of pollutants in ambient air and stationary sources. Raleigh, NC: EPA-600/9-84-00 (1983).
- [17] Busch KA, Leidel NA, Statistical Models for Occupational Exposure Measurements and Decision Making. In: Advances in Air Sampling, ACGIH. Chelsea, MI: Lewis Publishers, pp 319-336.
- [18] Kovein RJ, Hentz PA [1988]. A Digital Current Capacity Tester for Rechargeable Batteries. *Appl Ind Hyg* 3(2):51-54.
- [19] Okladek J [1988]. An Overview of Flow Metering Devices. *Amer Lab* 20:84-90.
- [20] Frazee PR, Tironi G [1987]. A Filter Cassette Assembly Method for Preventing Bypass Leakage. *Am Ind Hyg Assoc J*, 48:176-180.
- [21] ACGIH [1996]. 1996 TLVs® and BEIs® Threshold Limit Values for Chemical Substances and Physical Agents and Biological Exposure Indices. Cincinnati, OH: American Conference of Governmental Industrial Hygienists, pp. 45-48.
- [22] Higgins RLJ, Dewell P [1967]. A Gravimetric Size Selecting Personal Dust Sampler. In: Davies CN, Ed. Inhaled Particles and Vapors II, Oxford: Pergamon Press, pp. 575-586.
- [23] Feldman R [1997]. A Jarless Method for the Calibration of Cyclone Assemblies. Unpublished information, Occupational Safety and Health Administration, Cincinnati, OH.