



NIOSH Manual of Analytical Methods (NMAM), 5th Edition

# General Considerations for Sampling Airborne Contaminants

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DEPARTMENT OF HEALTH AND HUMAN SERVICES  
Centers for Disease Control and Prevention  
National Institute for Occupational Safety and Health



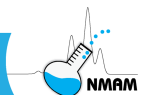


# 1 Choosing measurement methods and sampling media

In choosing methods for sampling of chemical and biological agents related to occupational exposures, thorough advance planning is required. Proper planning minimizes sampling and measurement costs while enabling the collection of high-quality data. Many criteria must be considered before collecting field samples from workplaces [ASTM International 2014]. The first step is to define the sampling objectives. These may include: a) documenting exposures in particular work settings; b) assessing compliance/non-compliance with existing regulatory or recommended occupational exposure limits (OELs); and/or c) determining sources of airborne contaminants. Sampling parameters that should be considered might include: a) type of sample (area vs. personal); b) contaminant(s) to be sampled; c) physical nature of airborne sample (vapor and/or aerosol); d) duration of sample collection; e) potential interferences; and f) estimated contaminant concentrations. Once these parameters are established, the suitable analytical method(s) and sampling media can be selected. For instance, when sampling for aerosols, the relevant particle size fraction to be sampled must be taken into account. Other general information needed to properly plan a sampling campaign include: a) the number of employees; b) the sampling strategy plan (discussed later); c) process flow diagram; d) safety data sheets on all process materials; and e) potential hazards involved in collecting and shipping field 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 measurement needs. Consultation with the laboratory 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 sections of the individual methods in NMAM can be helpful in choosing which of the available methods is best for a particular situation. Apart from NIOSH methods, methods from other organizations such as OSHA, ASTM International and ISO may be appropriate [Ashley 2015].

Whether through consultation with the laboratory or as cited in the relevant measurement method, the sampling media will be specifically identified. Methods will specify parameters such as: a) pore size and type of filter (for aerosol collection); b) concentration and amount of liquid media required (e.g., for impinger sampling or impregnation of filters); and c) type and amount of solid sorbent (for collection of gases / vapors); see below for common types and characteristics of various sampling media.



The physical state(s) 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 correct sampler consists of a membrane filter to collect particulate matter and a solid sorbent tube to trap the vapors of certain PAHs so that total collection is assured. Also, for sampling of gases and vapors, it is generally necessary to mount a prefilter in front of the gas/vapor collection media in order to trap aerosols that might otherwise interfere with subsequent analysis of target analytes.

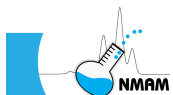
The sampling pump used to collect the sample must also be compatible with the sampling needs and the media used. Specifically, the pump must be capable of maintaining the desired flow rate over the time period needed using the sampling media specified. Some pumps may not be able to handle the large pressure drop of the media. This may be the case for fine 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 higher flow rates. As a rule of thumb, higher-flow pumps (>1 L/min) can handle at least 3 kPa pressure drop at 1 L/min for 8 h. Some pumps can handle up to 7.5 kPa pressure drop at flows up to 5 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 use.

## 2 Types and uses of solid sorbents\* [Melcher 1987]

### a. Activated charcoal

By far the most commonly used solid sorbent is activated charcoal. This sampling medium is characterized by a very large surface area to weight ratio. It has a reactive surface and 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 that 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 characteristics as well.

\*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).



## **b. Silica gel**

Silica gel is less reactive than charcoal. In part because of its polar nature, coated silica gel has been shown to be effective for sampling airborne ketones and aldehydes [Tejada 1986; García-Alonzo and Pérez-Pastor 1998].

## **c. Porous polymers**

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

## **d. Synthetic sorbents**

Synthetic carbonaceous sorbents demonstrate properties midway between charcoal and porous polymers.

## **e. Coated sorbents**

Coated sorbents are those 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 of interest [Kennedy 1988].

## **f. 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 [Langhorst 1983].

## **g. Thermal desorption**

Thermal desorption tubes may contain several different sorbents in order to collect a wide range of different chemical agents [Hodgson et al. 1988]. 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. Subsequent analysis is often by gas chromatography/mass spectrometry (GC/MS).



## 3 Types and uses of aerosol samplers

[Baron 1998]

### a. Membrane filters

By far the most frequently used as sampling media for aerosols are membrane filters. This class of filters includes those made from polyvinyl chloride, polytetrafluoroethylene (PTFE), copolymers, and mixed cellulose esters (MCE). Filters from this class are used for sampling airborne particles such as asbestos, crystalline silica, particulate PAH's, particles not otherwise regulated, and elements for atomic spectrometric analysis.

### b. Glass and quartz fiber filters

Quartz filters have replaced glass fiber in many applications. They are used in applications such as sampling for mercaptans and diesel exhaust. Impregnated quartz filters are often used to sample reactive aerosols.

### c. Polycarbonate straight pore filters

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

### d. Respirable dust samplers

Cyclone samplers are used to collect airborne respirable dust. For instance, a high-flow cyclone attached to a sampler containing a polyvinyl chloride filter is used to collect respirable crystalline silica.

### e. Inhalable dust samplers

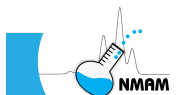
Various samplers have been designed and/or evaluated for collecting samples of inhalable airborne particles. For example, the Institute of Occupational Medicine's (IOM) sampler is used, in conjunction with a polyvinyl chloride filter, for sampling formaldehyde on dust [NIOSH 1994a].

## 4 Factors affecting the collection of gases, vapors, and aerosols

[Hebisch et al. 2009; Jones 1994; Baron 1994; Kulkarni et al. 2011]

### a. Temperature

Since adsorption is typically exothermic, adsorption is generally 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. The temperature stability of a filter must be considered when sampling in hot environments.

### b. 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., for chlorine and bromine [Cassinelli 1991] and formaldehyde [NIOSH 1994b]. This effect varies from substantial for more polar sorbents, such as charcoal and silica gel, to a smaller effect for synthetic carbonaceous and porous polymers.

Filter media may also be affected by humidity; moisture may affect a filter's collection efficiency. Very low humidities ( $\leq 10\%$  RH) may make some filters (e.g., MCE) develop high charge levels, causing non-uniform deposits and repulsion of particles [Chen and Baron 1996]. Water absorption by some filters (e.g., MCE) can cause difficulty in obtaining tare weights for gravimetric analysis; thus weight-stable filter materials must be used for this application.

### c. 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 [Foerst 1979]. The collection efficiency of membrane filters will change with face velocity.

### d. 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 [Foerst 1979]. The effect of concentration is similar for filters.

**\*\*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.



## e. Particle characteristics

Filter collection efficiency is a function of effective pore size [Lee and Ramamurthi 1993]. Particles smaller than about 0.2  $\mu\text{m}$  are collected primarily by diffusion, while particles larger than about 0.2  $\mu\text{m}$  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  $\mu\text{m}$  size range. Polycarbonate straight pore filters exhibit poor collection by diffusion, so particles smaller than the pores are not collected efficiently.

## f. Filter considerations

The pressure drop of a filter can limit the sampling time because of the load on the personal sampling pump [Breuer 2012]. In addition, pressure drop increases with dust loading on the filter [Lippman 1995]. Fine particles (<0.5  $\mu\text{m}$ ) will increase the pressure drop much faster than coarse particles (>10  $\mu\text{m}$ ). Heavy loading (> about 2 mg) may result in poor adhesion of collected particles to the filter surface.

# 5 Establishing sampling parameters

Once the sampling media and measurement method are chosen, the specific sampling parameters need to be determined [Eller 1986; ASTM International 2013]. 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 5 L/min for 8 h for most aerosols or 10 to 200 mL/min for 8 h for most sorbent tube samples. It is necessary to consider the applicable OEL for short-term (e.g., 15 min) or long-term (e.g., 8 h) time-weighted average (TWA) sampling. 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). 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.

## a. Sampling for gases and vapors using solid sorbents

NIOSH Method: 1453 for Vinyl Acetate [NIOSH 2013]

Recommended Sample Volume: 1.5 – 24 L

Applicable Range: 0.07 to 46  $\text{mg}/\text{m}^3$  (0.02 to 13.1 ppm)

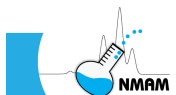
NIOSH REL: 14  $\text{mg}/\text{m}^3$  (4 ppm) - Ceiling

17.5  $\text{mg}/\text{m}^3$  (5 ppm) – (European OEL) TWA

Recommended Flow Rate: 0.05 to 0.2 L/min

Breakthrough Time: 30 min @ 0.2 L/min and 150  $\text{mg}/\text{m}^3$

Breakthrough Capacity: 9 mg



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

### 1.) Ceiling Determination:

If sampling were done at 0.2 L/min for 30 min and a total sample volume of 6 L collected, would this present a breakthrough problem? Probably not, since breakthrough tests were carried out in concentrations ca. 10x above the NIOSH Ceiling value [NIOSH 2013]. In the breakthrough test, a concentration of 10 times the NIOSH Ceiling value (150 mg/m<sup>3</sup>) was sampled at 0.2 L/min for 30 min (6 L) before breakthrough occurred, collecting a total weight of 9 mg of vinyl acetate. This test was conducted in a humid environment with only vinyl acetate present. A safety factor of 50% should normally be allowed to account for humidity effects. Thus, if sampling is done for about 15 min at 0.2 L/min, levels of vinyl acetate up to 40 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 sampling rate at 0.1 L/min might be done. With the safety factors built in, collecting a 6-L sample should be acceptable.

### 2.) TWA Determination:

In a similar situation, the goal is to collect 8-h samples for comparison to the 5 ppm TWA [NIOSH 2013]. If sampling were done at 0.1 L/min, then the total sample volume would be 48 L, substantially above the 24-L recommended sample volume. If the flow was dropped to 0.05 L/min, then the sample volume would be halved (to 24 L, the maximum recommended in the method). This sample volume might be acceptable if the vinyl acetate concentrations are around 10 ppm and no other competing organics are present, e.g., acetone. However, the safer approach would be to collect two consecutive samples at 0.05 L/min for 4 h (total sample volume of 12 L each).

## b. Pushing a method to the limit – limit of quantitation

NIOSH Method 1009 for Vinyl Bromide (VB) [NIOSH 1994c]

Recommended Sample Volume: <10 L @ 0.20 L/min or less

Working Range: 0.3 to 33 ppm (1.3 to 145 mg/m<sup>3</sup>) for a 6-L air sample;

this equals 8 to 355 µg VB per tube

Limit of Detection: 3 µg VB per tube

In this particular example, let us say that the object is to estimate exposure down to 0.1 ppm (0.44 mg/m<sup>3</sup>), 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 lower, 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 (with concomitant breakthrough a concern). In pushing a method to the limit, it is often necessary to sample beyond the breakthrough volume, normally 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 apply outside the limits tested.

### c. Sampling for aerosols using a filter

NIOSH Method 7908: Non-volatile Acids (Sulfuric and Phosphoric Acids)  
[NIOSH 2014]

Recommended Sample Volume: 15 – 2000 L

Applicable Range of the Method: 0.010 to > 10 mg/m<sup>3</sup>

NIOSH REL: 1 mg/m<sup>3</sup> (H<sub>3</sub>PO<sub>4</sub>) (TWA); 3 mg/m<sup>3</sup> (STEL)

Recommended Flow Rate: 1 to 5 L/min

Suppose it is desired to determine both an exposure taking place during a specific 15-minute operation as well as a TWA exposure of workers exposed to phosphoric acid, and the concentrations are unknown.

#### 1.) 15-Minute Process Sample:

This sample would meet the method conditions by sampling for the 15 minutes at 5 L/min, since this would collect 75 L. Sampling at 1 L/min for 15 minutes would probably not allow for the collection of sufficient sample required for analysis.



## 2.) TWA Determination:

In this situation, it is necessary to collect an 8-h sample to compare with the  $1 \text{ mg/m}^3$  NIOSH REL. Since an 8-h TWA sample covers 480 minutes, sampling can no longer be done at 5 L/min since this would collect >2000 L, in excess of 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.

## 6 Bulk samples

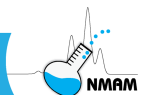
The collection of bulk samples can often assist with air sampling efforts. This is especially true where there is mixed solvent exposure or unknown dust exposure, and for determining silica content of dusts. The primary purpose of obtaining bulk samples is to provide the analytical laboratory with a large enough sample for qualitative and sometimes quantitative analysis. The two main 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 a 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 sorbent tube 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 identifying which substances are present rather than their exact concentrations (the latter aim is accomplished through the separate collection of air samples in accordance with defined method parameters). 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 sampling, either a bulk air or solid bulk sample (e.g., a deposited 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 identify the substances present in the workplace and, in some cases, to estimate 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 whether a certain contaminant of interest is present, e.g., benzene. In some cases, up to 30 solvents may be present, but their identities and proportions are not certain. This example is also true for dusts, as was discussed previously for silica, and for metals, which may exist in trace quantities.



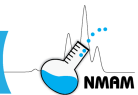
In choosing bulk samples for collection, the end goal must be considered: qualitative and/or quantitative analysis. Any information that can be given to the laboratory on what may or may not be present will help with the analysis. Advance consultation with the laboratory is desirable. In choosing bulk 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 locations near the workers' job site. In other cases, a process dust sample may be chosen to determine the composition of the material before it becomes airborne.

When shipping bulk samples, care must be taken to preserve the integrity of the samples and to follow established shipping regulations. If applicable, hazardous shipping procedures must be followed. Consult with an experienced hazardous goods shipper to determine appropriate protocol. Only 5 to 10 mL of the liquid or 10 to 100 mg solid is typically needed, so generally bulk sample sizes are kept small. It is important to consult with the laboratory before collecting bulk samples to ensure proper sample size and containers. 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. 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 volatile compounds together with air samples. Specific labeling is usually required when dry ice is used in shipping.

## 7 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 the same way as the air samples, except that no air is drawn through them. Media blanks are simply unopened, new samplers which are sent to the laboratory with the field 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, and 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 using careful sampling protocols. The general recommendation for the



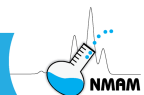
number of field blanks is three field blanks for each batch of samples. Media blanks should also be included. These unexposed, unopened samplers are used to give an estimate of media background. The laboratory should analyze at least three (3) media blanks from the same lot as the field samples. This number should be increased for media which are coated or impregnated with reagent. Again, it is crucial to consult the specific method for the number and type of blanks as these numbers will vary. Another recommended practice is to include blind spiked samples as quality control checks of the analytical laboratory.

## 8 Direct-reading methods [Pearce and Coffey 2011]

The variety of types of direct-reading methods available is large and expanding, including detector tubes (both short- and long-term), aerosol monitors, passive monitors for certain gases and portable instrumentation for gas chromatography or infrared spectrometry [Todd 1997]. Many direct-reading instruments now used for personal or area measurements have evolved from laboratory or process control instruments [Woebkenberg and McCammon 1995; Todd 1997]. Some direct-reading instruments are screening devices while others give quantitative result and can be used for compliance purposes [Song et al. 2001; Ashley et al. 2002].

Some of the considerations (e.g., specificity and sensitivity) for the use of direct-reading methods for quantitative determinations are similar to those already given for filter or sorbent sampling and analytical 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 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, i.e., that of field calibration of the direct-reading instrument, falls on the field sampling personnel.

\*\*\***NOTE:** Portable instrument are generally described as weighing less than 5 kg and powered by self-contained batteries [Ashley 2003]. For personal monitoring, the instrument configuration should be such that the breathing zone can be monitored.



## 9 Sampling Strategy

To obtain the maximum amount of information during the course of a sampling campaign with a minimum number of samples, a statistical sampling strategy should be developed before conducting any study [Leidel et al. 1977; ASTM International 2013]. 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 occupational exposures.

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 the scheme below. (Other figures are applicable 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 1. Minimum sample size (n) for including (@ 90% confidence level) at least one high risk employee<sup>A</sup> [Leidel et al. 1977]**

Size of employee group (N)																			
1	2	3	4	5	6	7	8	9	10	11-	13-	15-	18-	21-	25-	30-	39-	40-	50+
										12	14	17	20	24	29	37	29	49	
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

<sup>A</sup> Exposure in highest 10% of N.

## 10 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 NIOSH methods. Consult individual methods for details regarding 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. Ordinarily, pumps should be calibrated in the laboratory and the field, both before field use and after each field sampling campaign.



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 [Okladek 1988]. Several electronic soap-bubble calibrators and dry-cell calibrators 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 calibration device is used, equivalent procedures should be followed.

- 1.) Set up the apparatus as shown in Figure 1.
- 2.) Ensure 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 operation; or 2) test the battery independently of the pump using a current capacity tester [Kovein and Hentz 1988]. 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 of water.
- 5.) Start a soap bubble in the buret and, with a stopwatch, measure the time 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 [Okladek 1988]. The expression for this correction is as follows.

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

$V$  (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)

$P_s$  = pressure of air sampled (same units as  $P_c$ )

$T_c$  = temperature during calibration of sampling pump (K)

$T_s$  = temperature of air sampled (K).

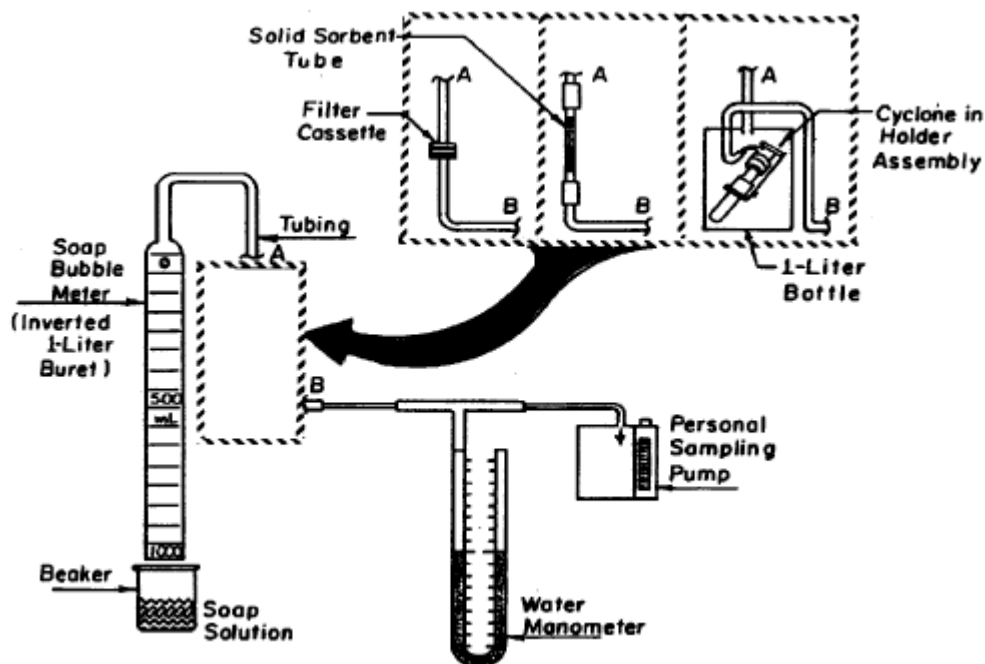
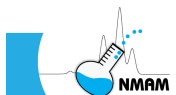


Figure 1. Calibration Apparatus

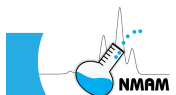
### b. Sampling instructions for solid sorbent tube sampler

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. 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. A minimum of three field blanks are normally required for each batch of samples.
- 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 a minimum of three 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.
- 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 and three additional tubes so that desorption efficiency studies can be performed on the same lot of sorbent used for sampling.



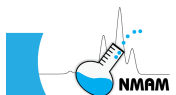


- 9.) Ship bulk samples in a separate package from the air samples to avoid contamination of the samples. If applicable, hazardous shipping procedures must be followed. Consult with an experienced hazardous goods shipper to determine appropriate protocol. Suitable containers for bulk samples are glass with a polytetrafluoroethylene (PTFE)-lined cap, e.g., 20-mL glass scintillation vials. It is important to consult with the laboratory before collecting bulk samples to ensure use of proper sample size and containers.

### c. Sampling instructions for filter sampler

Use these instructions for personal sampling of inhalable 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 a filter or internal capsule in a cassette filter holder. Support the filter by a cellulose backup pad or stainless steel screen. Close the filter holder to ensure that its parts mate evenly and securely to prevent leakage [Frazee and Tironi 1987]. Seal the filter holder with plastic tape or a shrinkable cellulose band.
- 3.) Remove the filter holder plugs and attach the filter holder to the personal sampling pump with a piece of flexible tubing. Position the filter holder in the worker's personal breathing zone, with the sampler inlet pointed downwards.
- 4.) Prepare the field blanks at about the same time as sampling is begun. These field blanks should consist of unused filters (or internal capsules) 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. Three field blanks are required for each batch of samples.
- 5.) Sample at the prescribed flow rate (usually 1 to 5 L/min) until the recommended sample volume is reached. Set the flow rate as accurately as possible (e.g., within  $\pm 5\%$ ) using a calibrated flowmeter. Take three replicate samples (minimum) for quality control for each set of field samples.

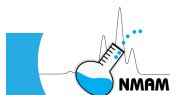


- 6.) Observe the sampler frequently and terminate sampling at the first evidence of excessive filter loading or change in personal sampling pump flow rate. (It is possible for a filter to become plugged by heavy particulate loading or by the presence of oil mists or other liquids in the air.)
- 7.) 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. Record the type of personal sampling pump used and location of the 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 chemically inert container. Never store, transport or mail bulk samples in the same container as the samples or field blanks. In addition to the samples and field blanks, ship at least four unused filters or filter capsules from the same lot for use as media blanks.

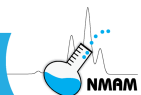
### **d. Sampling instructions for filter + cyclone sampler**

Use these instructions for personal sampling of respirable (or thoracic) aerosols [Frazee and Tironi 1987]. Methods requiring these instructions specify CYCLONE + FILTER as the sampling method.

- 1.) Calibrate the pump to the rate specified by the 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 cassette filter holder. Use a conductive or static-dissipative cassette. 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.) Position the cyclone assembly in the worker's personal breathing zone and attach the personal sampling pump to a belt or harness. Ensure that the cyclone hangs vertically with the inlet pointed downwards. Explain to the worker that 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. A minimum of three field blanks are required for each batch of samples.
- 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. 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 a minimum of three 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, sr,



calculated from these replicates, should be recorded on control charts and action taken when the precision is out of control.

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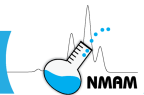
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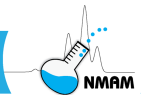
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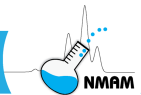
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