NMAM

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Factors Affecting Aerosol Sampling

by Paul A. Baron, Ph.D., NIOSH

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1 Introduction

The need for aerosol sampling is driven by research or regulatory needs to understand or quantify the properties of airborne particles in the workplace or ambient environments. The property of most common interest is the airborne concentration of particulate mass defined as the aerosol mass per unit volume of air, usually expressed in units of micrograms or milligrams per cubic meter. Alternatively, concentrations of other related properties such as surface area or number, or particle size distributions are also of interest in certain cases, especially where exposure to nanoparticles or ultrafine aerosols are involved. In many applications, airborne concentration of a certain chemical or analyte, usually expressed in terms of micrograms per cubic meter, is more important. Aerosol sampling is the process of collecting a representative sample of airborne particles of interest from the air environment by physically separating them from the sampled air of known volume. The degree to which the physically separated sample represents the in situ aerosol depends on the design of the physical separation device, often known as the aerosol sampler. Other factors that affect the representativeness of the particulate sample include environmental conditions (e.g., wind, temperature, humidity), particle characteristics (particularly if they are highly irregular or nonspherical), and subsequent analytical methods used for particle analysis. This chapter focuses mainly on the key characteristics of aerosol samplers that may influence the representativeness of the sampled aerosol. Direct-reading aerosol samplers are not discussed in this chapter.

Most samplers use size-selective inlets that conform to certain health-based conventions. The American Conference of Governmental Industrial Hygienists (ACGIH) [Vincent 1999a; ACGIH 2015], the International Organization for Standardization (ISO) [ISO 1995], and the European Standardization Organization (Comité Europeén de Normalisation, CEN) [CEN 1993] have adopted identical particle size-selective sampling conventions for inhalable, thoracic, and respirable aerosols (Figure 1). The purpose of these conventions is to provide a scientific basis for a new generation of particle size-selective occupational exposure limits (OELs) for aerosols. Such OELs can therefore be matched to the relevant sites of aerosol deposition after inhalation into the respiratory tract, and in turn to the health effects of interest in a given exposure assessment. These sampling conventions are used throughout this manual unless otherwise specified.

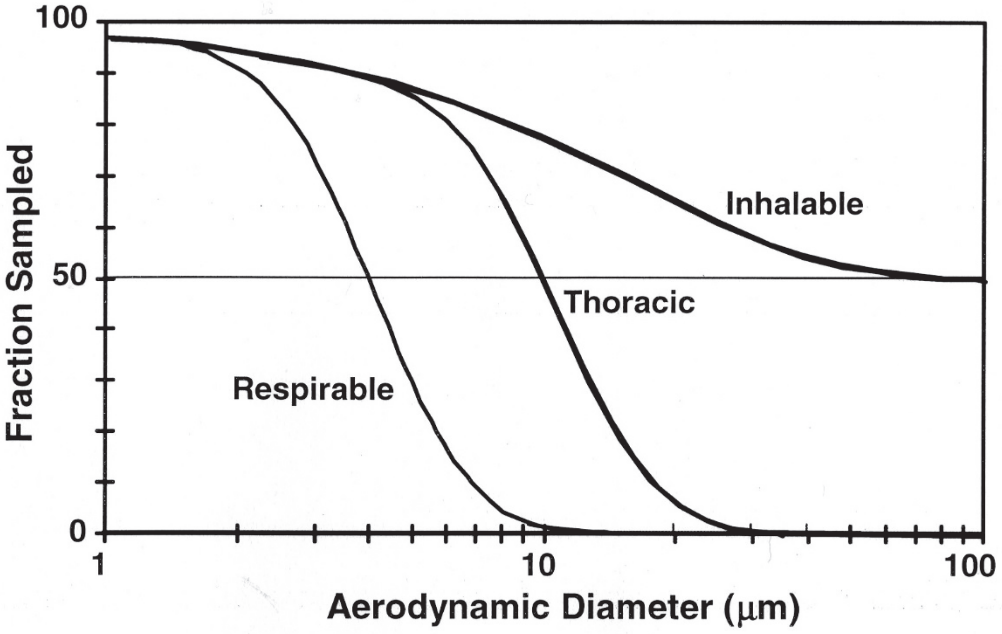

Figure 1. ISO/ACGIH/CEN sampling conventions [ISO 1995]. An ideal sampler should have a sampling efficiency curve that matches one of these curves as closely as possible under all wind directions and velocities. The 50% cut points for the respirable and thoracic conventions are 4 and 10 µm, respectively.

The criteria presented in this manual are used to determine the most appropriate aerosol sampling equipment. The type of sampling to be performed determines which criteria are important for estimating the adequacy of the sampler and determining aerosol concentration levels. For example, “total dust” samplers generally do not have a size selective particle classifier preceding the filter media and fall under the inhalable sampling convention. Alternatively, sampling for regulatory or voluntary compliance with aerosol exposure standards usually requires greater accuracy, increased efficiency, size-specific selectivity, and good analytical precision. Furthermore, regulations may require the use of a specific sampler and sampling conditions to standardize sampling results (eliminate bias) and reduce uncertainty among laboratory reports. See the chapter on measurement uncertainty and NIOSH method accuracy for further discussion on standardization and aerosol measurement error.

Open-face filter cassettes do not use constricted opening or tubing and expose the filter directly to the aerosol to minimize the losses. They provide relatively uniform particle deposition on the filter. On the other hand, closed-face filter cassettes are often necessary to connect to upstream tubing or size-selective inlets.

Over the past decades, researchers have pointed out strengths and weaknesses with several types of aerosol samplers (Figures 2A-J). Some of these samplers were adapted from existing devices used for other purposes, e.g., the 10-mm nylon cyclone and the 37-mm cassette, without the benefit of current testing technology and understanding of particle behavior.

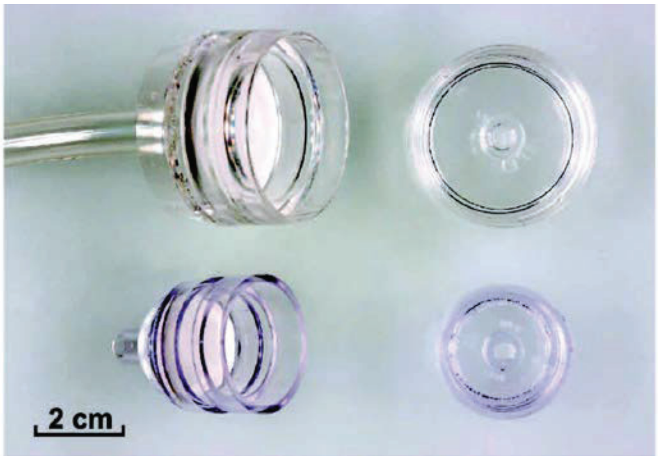

Figure 2A. 37-mm (top) and 25-mm (bottom) filter cassettes. Open faced cassettes are shown on left. Closed-faced cassettes include placement of clear piece (on right) over filter. These are shown in acrylic copolymer (clear, non-conducting) material and are more prone to electrostatic losses. Other construction materials are available, including conducting plastic. Sampling flow rates range from 0.5 to 10 L/min.

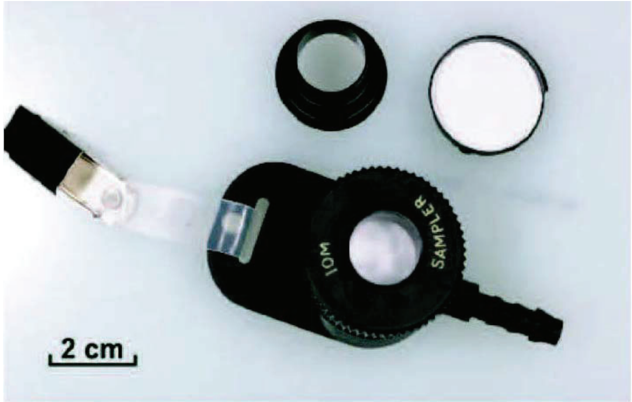

Figure 2B. IOM inhalable sampler. This is the first sampler designed specifically to match the inhalable sampling convention. The sampling cartridge is shown on the right with the inlet, filter, and support grid. All dust entering the cartridge is collected and analyzed. Sampler is made of conductive plastic and operates at 2 L/min.

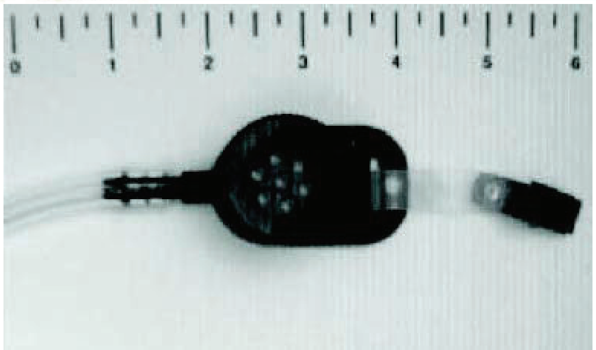

Figure 2C. Seven hole sampler used for inhalable dust sampling in the UK. Sampler is made of conductive plastic and operates at 2 L/min.

Figure 2D. Button sampler. So-named because the inlet, a hemispherical screen with ~380 µm diameter holes, resembles a large button. It was developed as an inhalable sampler with reduced wind direction response and improved filter deposit uniformity. The sampler uses metal construction and operates at 4 L/min.

Figure 2E. Asbestos sampling cassette. The long (50 mm) inlet was designed to prevent incidental contact with the filter surface and, when facing downward, acts to a certain extent as an elutriator, preventing larger particles from reaching the filter surface. It is made of conductive plastic and is operated at a flow rate between 0.5 L/min to 16 L/min.

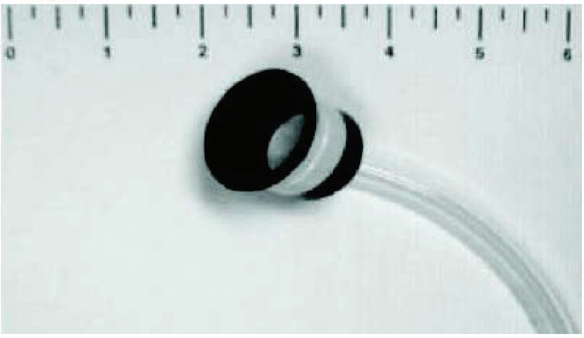

Figure 2F. Bell mouth cowl sampler. An alternative to the standard asbestos sampling cassette. The inlet is flared to reduce the effect of external air motion on sample uniformity. It is made of conductive plastic and is operated at a flow rate between 0.5 L/min to 16 L/min.

Figure 2G. Dorr-Oliver 10-mm nylon cyclone. It is used as a respirable sampler most often at 1.7 L/min. A version of this sampler is used at 2 L/min in coal mines with a 1.38 correction factor applied to the resultant mass. The holder encases body of the cyclone so that only the inlet section is visible just below the connection to the 37 mm cassette. The coal mine dust sampler version uses a cassette with an aluminum cartridge encasing the collection filter.

Figure 2H. Higgins-Dewell cyclone. This sampler was developed and used primarily as a respirable sampler in the UK. This version provides an interface directly to a 37-mm cassette. Cyclone construction is steel and operates at 2.2 L/min.

Figure 2I. GK2.69 cyclone. This cyclone was designed to match the slope of the thoracic and respirable conventions more closely than other sampling cyclones. Cyclone construction is stainless steel and operates at 4.2 L/min as a respirable sampler and 1.6 L/min as a thoracic sampler. GK4.162 RASCAL Cyclone is also available for higher flow rates of 8.5-9 L/min.

Figure 2J. Personal cascade impactor sampler (model Marple 290, Thermo Fisher Scientific). The sampler has five stages in the version shown and an additional three stages are available to provide cutpoints from 0.4 µm to 21 µm. The flap over the inlet reduces direct projection of large particles into the inlet. This sampler allows measurement of aerosol size distribution and calculation of respirable and thoracic fractions. It is constructed of aluminum and operates at 1.7 L/min.

More recently developed aerosol samplers were designed to either maximize the information gained regarding aerosol concentration levels or to minimize any inherent losses associated with the sampler design. Thus, each sampler design may have been based on some of the following criteria: inlet or aspiration efficiency, classifier accuracy, cassette assembly (bypass leakage), electrostatic losses, particle deposition uniformity, collection media stability, sampler surface losses, and sampler field comparisons. These criteria are discussed in the following sections of this chapter.

2 Inlet efficiency of the sampler

An important review of sampling theory and practice was compiled in a book by Vincent [2007]. The inlet efficiency of several samplers has been evaluated including thin-walled tubular inlets [Grinshpun et al. 1993], a cyclone [Cecala et al. 1983], an asbestos sampler [Chen and Baron 1996], total aerosol sampling cassettes [Fairchild et al. 1980], and inhalable aerosol samplers [Kenny et al. 1997; Aizenberg et al. 2001]. All samplers have an inlet efficiency, also called aspiration efficiency, which varies as a function of particle aerodynamic diameter, inlet velocity, inlet shape and dimensions, dimensions of the body it is attached to, external wind velocity, and external wind direction. The *aspiration efficiency* of an aerosol sampler can be defined as

$$A = \frac{C_S}{C_0}$$

where C_S is the concentration of particles passing directly through the plane of the sampling orifice and C_0 is the ambient concentration. This is true for aerosol samplers for which the entire amount of aerosol that enters the plane of the sampling orifice is quantified, as is the case for the IOM sampler (SKC, Inc., Eighty Four, PA). However, it is important to note that many commercially available aerosol samplers use only the aerosol collected on the filter that is housed in the sampler, while any particles deposited on the inner surfaces upstream of the filter are disregarded. Performance of this type of sampler is therefore best characterized by its *sampling efficiency*, in which the aspiration efficiency is modified by the particle size-dependent wall losses prior to the filter. The aspiration or sampling efficiency of a particular aerosol sampler, whichever is the most relevant, expressed as a function of particle aerodynamic diameter, is the primary index of sampler performance. The overall size-dependent transmission efficiency of the sampler must match the appropriate health-based criterion (e.g., the inhalability criterion) to allow its use for a health-based assessment of personal aerosol exposure.

As an aerosol is being sampled, the large-particle trajectories are more affected by external flow fields than those of small particles. Thus, the shape, orientation, and inlet flow field will be most critical for inhalable aerosol sampler inlets; they will be less important for thoracic samplers and unlikely to be important for respirable samplers, except at very high wind velocities. The flow field near the inlet of a sampler is different when the sampler is mounted on a person (or in laboratory simulations using a mannequin) than when it is freestanding. Therefore, it has been recommended that measurement of inhalability be determined from mannequins in wind tunnels [Vincent 1999a; Kennedy et al. 1995; Kennedy and Hinds 2002; Aitken et al. 1999]. Flow field studies using mannequins in wind tunnels indicate that the air is slowed down by the body, resulting in an enrichment of large particles in the upstream side of the body [Rodes et al. 1995]. When intended for use at low wind speeds (less than about 1 m/s [200 ft/min] (representing most indoor workplaces), it may be possible to test the samplers as freestanding devices if it can be shown there is no effect from the mannequin body [CEN 1997]. However, if the sampler is to be used at higher windspeeds, which frequently occur outdoors, personal aerosol samplers should be evaluated on a mannequin in a wind tunnel [CEN 1997].

Respirable aerosol samplers generally do not have problems with inlet effects because the particles being sampled have low enough inertia and settling velocity. However, Cecala et al. [1983] found that the 10-mm nylon cyclone operated free-standing in a wind tunnel oversampled at external air velocities greater than 4 m/s when the inlet faced the wind and undersampled at 90° and 180° to the wind at velocities greater than 1 m/s. The maximum sampling error of about 40% was observed at 10 m/s. It is expected that these errors would be reduced if the sampler were located on a person, because the air velocity decreases near the body surface. It should be noted that the Cecala et al. work was conducted in the context of aerosol sampling in underground mining environments. Here, windspeeds of the magnitude quoted are not uncommon. However, in industrial workplaces more generally, windspeeds are much lower. Two surveys of a wide range of workplaces [Baldwin and Maynard 1998; Berry and Froude 1989] revealed that actual indoor windspeeds rarely exceeded 0.2 to 0.3 m/s and more typically were less than 0.1 m/s.

The EPA PM_{10} standard for environmental sampling specifies a sampler that has a 50% cutpoint at 10.6 µm particle diameter, approximately the same as that for the thoracic sampler [Baron and John 1999]. Although the requirements for environmental PM_{10} samplers stipulate wind tunnel testing, similar work on personal PM_{10} and thoracic samplers is yet to be performed. It is expected that these samplers will be more susceptible to wind effects than respirable samplers because larger particles are more susceptible to inertial and gravitational effects.

The most extensive comparison of available inhalable aerosol samplers was that carried out under the auspices of the European Commission (EC) [Kenny et al. 1997]. Eight samplers were tested: CIP-10 (foam-based, French); 37-mm closed-face cassette (Spain and US) (Figure 2A); 37-mm open-face cassette (Sweden) (Figure 2A); PAS-6 (Netherlands); PERSPEC (Italy); GSP (Germany, sold as CIS sampler in US; BGI, Inc. Waltham MA); IOM (United Kingdom; (Figure 2B); and the Seven-Hole Sampler (United Kingdom; Casella CEL, Inc., UK) (Figure 2C). Conditions of the experiment included measurement of sampler collection efficiencies on a mannequin for aerosol particles with diameters as large as 100 µm at a wind speed of 0.5m/s, 1.0 m/s, and 4 m/s. Samplers were positioned on a mannequin rotating within a wind tunnel. The samplers were all conductive; the 37-mm cassette samplers were painted with an external conductive coating. The aerosol was, however, not neutralized. The results of this experiment indicated high inter-sampler variability, but permitted estimates of bias relative to the inhalable convention. The EC study also indicated that most samplers work reasonably well at low wind speeds (<1 m/s) for particle median diameters below 25 µm [Kenny 1995]. The study indicated that experiments of this type were difficult, expensive, and generally had poor precision. Perhaps better understanding of the flow field near the body may lead the way to improved and simplified sampler testing. Recent work suggests ways of making the wind tunnel testing of inhalable samplers simpler and less expensive, e.g., by using a compact body to simulate the chest of a mannequin [Witschger et al. 1997] and by using miniaturized mannequins and samplers that are calculated to be aerodynamically equivalent [Ramachandran et al. 1998].

The orientation and diameter of an inhalable sampler inlet may affect the collection of very large particles (generally >100 µm), since these may be thrown into the inlet as projectiles. The current definition of inhalable aerosol only covers particles up to100 µm aerodynamic diameter.

In situations where large particles can be generated (e.g., abrasive blasting, wood working, and grinding operations) excessive collection of particles up to the millimeter range is likely to occur. There have been some attempts to modify inlets with shields to provide a barrier against the collection of large particles, but these modified inlets have not been demonstrated to provide the same agreement with the inhalable convention as the unmodified ones.

Another potential problem with inhalable samplers is the collection of passively sampled particles. Measurements when the sampler airflow is turned off indicate that IOM samplers, which pointed outward from the body and had a large inlet diameter (15-mm), can collect quite significant amounts of dust, with median values of 9 to 32 percent of the mass collected during active sampling [Lidén et al. 2000b]. Open-faced cassettes had only 2 to 11 percent of the mass passively collected. These samplers have a larger inlet (37-mm), but point downward, reducing the likelihood of particle settling onto the collection surface. The mechanism of collection is unclear, but the dust may be transported into the inlet by turbulence and deposited by settling or turbulent diffusion. How this passively collected dust modifies the amount collected during active sampling remains under investigation.

A comparison of measurements obtained with the 37-mm closed-face cassette (4-mm inlet diameter) to the IOM sampler (15-mm inlet diameter) in several workplaces gave similar results when the material on the interior walls of the 37-mm cassette were added to the analyte deposited on the filter [Demange et al. 2002; Harper and Demange 2007]. This suggests that the two samplers can have similar inlet efficiencies in spite of differences in inlet size and orientation if the median particle size sampled is not too large. Therefore, if the total aspiration (which includes the mass from filter and the wall deposits) of the IOM sampler conforms with the ISO inhalable size-selection criterion, then so does the total aspiration of the closed cassette filter. Several studies have now shown that in metals industries the total of mass from particulate filter and the wall deposit are comparable for both the closed cassette filter and the IOM samplers [Harper and Demange 2007].

3 Classifier accuracy

The theory of classifier separation is based on particle aerodynamic diameter, which is defined as the diameter of a 1 g/cm^3 density sphere having the same gravitational settling velocity as the particle in question. If the particle is markedly nonspherical or irregularly shaped, the aerodynamic diameter may depend on particle's orientation and other factors, possibly contributing to sizing errors. For example, fibers and plate-like particles settle slightly differently depending on orientation [Kulkarni et al. 2011]. Thus, the sampling conventions, based on aerodynamic diameter of particles reaching specified parts of the respiratory system, become somewhat ambiguous for these types of particles. For such nonspherical particles, further testing of classifiers to simulate particle behavior in the respiratory tract may be necessary. For instance, Maynard [1996] found that plate-like particles may orient differently in elutriators, impactors, and cyclones. This preferred orientation in a cyclone produced a collection efficiency 15% below that estimated to occur in the respiratory system. In addition, Baron et al. [2008] showed that the overall enveloping physical size of airborne single-walled carbon nanotube (SWCNT) agglomerates is much larger than their aerodynamic size, by a factor of up to 10. Ku and Kulkarni [2015] measured both aerodynamic and mobility (or diffusion) diameters of airborne carbon nanotubes (CNTs) and other nanomaterials to show that aerodynamic diameter is smaller by a factor of 2 to 4 than mobility diameter for SWCNT and multi-walled CNT particles. These studies indicate that relevant equivalent diameters must be used to obtain reliable estimation of lung deposition fraction. Improved understanding of fiber [Esmen and Erdal 1991], nanotube [Baron et al. 2008; Ku and Kulkarni, 2015] and plate-like particle [Maynard 1996] behavior in the respiratory tract is needed to aid in development of more accurate samplers for these types of particles. The phase (i.e. liquid or solid) of the aerosol particles also influences sampling errors. Koehler et al. [2012] examined the sampling efficiency as a function of particle phase of three personal aerosol samplers, including the IOM and button sampler. They found that large liquid droplets have low transmission efficiencies through the screened inlets and that the bounce of solid particles significantly affects the aspiration efficiencies of screened inlets.

Various types of classifiers have been constructed to meet the ACGIH/ISO conventions. For example, respirable samplers have used cyclones [Caplan et al. 1977], impactors [Marple 1978; Kimura 1978; John 1994], elutriators [Lynch 1970], and porous foam [Brown 1980; Courbon et al. 1988] to remove non-respirable particles from the aerosol prior to filter collection. The technology for testing these samplers has improved in recent years through use of a real-time aerodynamic sizing instrument and resulted in quicker and more precise measurements [Baron 1993; Gudmundsson and Lidén 1998]; this technique has allowed the accuracy of these samplers to be investigated more carefully [Bartley et al. 1994]. However, a round-robin comparison of 50% cut-point measurements from six laboratories using an aerodynamic sizing instrument to test the same cyclone agreed within a range of 11% [Lidén 2000a]. Further work on the testing protocol is needed to improve interlaboratory agreement. Many current classifiers do not match the shape of the respirable convention exactly and produce biases that depend on size distribution. Two comparisons of several respirable samplers have been performed using the aerodynamic sizing technique [Chen et al. 1999b; Görner et al. 2001].

The introduction of the thoracic fraction in the ACGIH/ISO conventions has spurred interest in thoracic classifiers for certain types of aerosols, e.g., cotton dust, asbestos and sulfuric acid [Baron and John 1999]. The performance characteristics of the vertical elutriator (operated at 7.4 L/min) used for cotton dust approximately meets the thoracic definition [Robert 1979]. Laboratories in many countries perform asbestos fiber measurement using the technique of counting only fibers with diameters of 3 µm or less; this size selection was shown to be approximately equivalent to thoracic sampling [Baron 1996]. Further tests indicate that several thoracic samplers may be appropriate for asbestos sampling [Jones et al. 2001; Maynard 1999]. Thoracic sampling is also recommended for sulfuric acid [Lippmann et al. 1987] and metal working fluids [NIOSH 1998].

Several samplers based on inertial, cyclone and foam separators have been specifically developed to meet the thoracic definition [Fabriès et al. 1989; Fang and Lippmann 1995; Mark et al. 1988; Kenny and Gussman 1997]. The CIP-10 sampler has been used for thoracic sampling in Europe [Gorner et al. 1994], but is not applicable to aerosols with a significant submicrometer fraction [Fabriès et al. 1989]. Several of these samplers have been tested to compare with the thoracic convention [Jones et al. 2001; Maynard 1999]. The GK2.69 cyclone (Figure 2I) has been used for metal working fluids [NIOSH 1998] and GK4.162 cyclone has been used for measurement of crystalline silica [Qi et al., 2015]. Several developmental samplers have also been developed. Koehler and Volckens [2013] have developed multistage regional deposition sampler that allows estimation of regional deposition of aerosol in the human respiratory system. This sampler is not suitable for gravimetric analysis but is well suited for measurement using variety of chemical analyses. A personal nanoparticle respiratory deposition sampler was developed by Cena et al. [2011] for particles smaller than 300 nm diameter, whose aspiration efficiency curves matches the fractional International Commission on Radiological Protection (ICRP) deposition curve for human respiratory tract below ~300 nm. Tsai et al. [2012] have developed a personal nanoparticle sampler which simultaneously collects both respirable and nanoparticles fraction (<100 nm aerodynamic diameter).

The PM_{10} standard for environmental sampling is very similar to the thoracic convention and impactors with a 10 µm cutoff size have been used for personal PM-10 sampling [Buckley et al. 1991]. A cascade impactor, e.g., the Andersen personal cascade impactor (Figure 2J), can be used to calculate the thoracic fraction of an aerosol. Although a thoracic sampler is commercially available (Figure 2I), further work is needed to determine its applicability for specific types of aerosol. For example, a thoracic sampler for fibers must result in a uniform deposit of the particles on the filter for accurate analysis results.

The overall accuracy of a classifier with respect to sampling in accordance with the one of the sampling conventions can be estimated using a bias map (Figure 3). The bias map displays the percent difference between the predicted mass collected by the sampler and the mass expected according to the convention as a function of the parameters of a lognormal particle diameter distribution for a range of likely workplace distributions. Such a bias map can be used for selecting a sampler for a workplace having a certain range of particle sizes or for developing samplers that agree more closely with the sampling conventions.

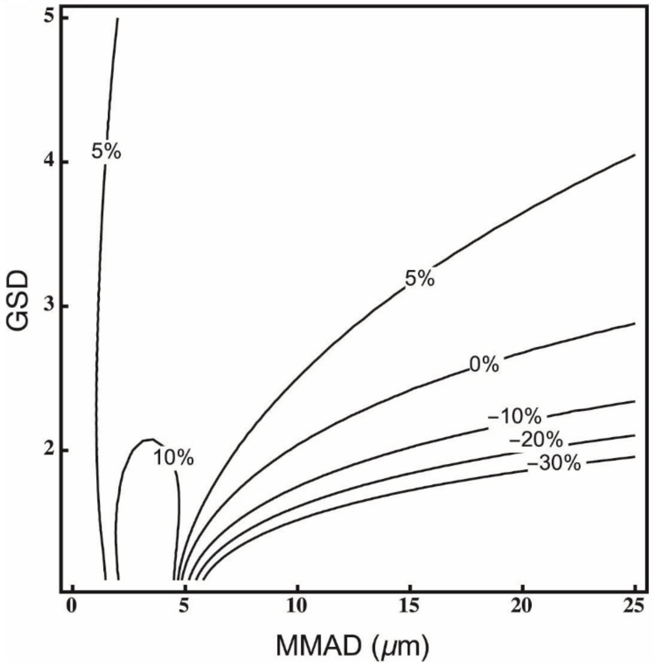

Figure 3. Bias map for the 10-mm nylon cyclone (operated at 1.7L/min) compared with the respirable convention [Courbon et al. 1988]. The contour lines represent percent bias for specific lognormal size distributions. The calculation of this map is based on laboratory measurement of cyclone penetration and can be used with field size distributions to estimate sampling bias.

The bias map in Figure 3 was created by: (a) fitting the penetration curve for the 10-mm nylon cyclone (Figure 2G) [Gudmundsson and Lidén 1998] at 1.7 L/min with a lognormal curve (a logistic curve also can be used), (b) calculating the bias between the respirable convention and the curve from the previous step for a range of lognormal size distributions, and (c) plotting the bias contour lines as a function of the size distribution mass median aerodynamic diameter (MMAD) and geometric standard deviation (GSD). The 10-mm nylon cyclone shows significant negative biases, especially at large MMADs and small GSDs, because the cyclone penetration curve drops off more rapidly with size than the curve for the respirable convention. The "best" flow rate to use in a workplace when sampling according to one of the conventions becomes a matter of judgment, depending on the size distribution typically encountered in that workplace. A cyclone that fits the convention more exactly will exhibit smaller biases throughout the entire size distribution range. Bias maps are available for several respirable samplers [Chen et al. 1999b; Görner et al. 2001]. It should be mentioned that in some cases, e.g., coal mine dust sampling, a single sampler is specified by regulation. This sampler specification eliminates the question of bias for that type of measurement.

Investigation of the effect of changing the physical dimensions of a commercial cyclone resulted in modifications that improved the match to the respirable sampling convention

[Lidén and Gudmundsson 1996]. Chen and coworkers developed a virtual cyclone that appeared to give excellent compliance with the respirable curve [Chen et al. 1999a]. It is possible to make samplers that have predicted biases less than 10% over the entire range of likely workplace size distributions. While the behavior of certain samplers, such as impactors, can be predicted theoretically, it is still important to measure penetration curves experimentally to ensure correct application of the theory. Bias maps based on these data then allow estimation of accuracy for a specific workplace application. As improved samplers are tested and become commercially available, more accurate thoracic and respirable aerosol measurement on a routine basis will be possible.

As mentioned above, several inhalable samplers were investigated in a wind tunnel to evaluate their sampling efficiency compared to the inhalable convention [Kenny et al. 1997]. Based on these data, sampler performance (maximum bias confidence limit) was ranked [Bartley 1998] and the IOM, GSP, and CIP-10 samplers were rated the best.

As interest in the new particle size-selective conventions by standards setting bodies has grown, efforts have been made to define protocols to guide the testing and validation process for available samplers. One approach was developed by the CEN [Lidén 1994; CEN 1998]. In the CEN model, for any given sampler to be tested, the first step is a critical review of the sampling process for the instrument in question. This is intended to identify factors that may influence the performance of the sampler, including particle size, windspeed, aerosol composition, filter material, etc. This is essential in the process of sampler evaluation, determining under what conditions the sampler will need to be tested. Three options are then presented for the testing of samplers: (a) the laboratory testing of samplers to compare performance with the sampling conventions, (b) the laboratory comparison of instruments, and (c) the field of comparison of instruments. Research projects have been conducted in recent years to define testing protocols (option a), funded both by the European Community and by NIOSH, to consolidate the scientific basis for such protocols and to identify improved and more cost-effective methods.

4 Sampler assembly

Some samplers are designed such that improper assembly can result in internal leakage, i.e., aerosol particles bypassing the filter. This bypass leakage has been noted in the 37-mm closed-face cassette [Frazee and Tironi 1987; Van den Heever 1994]. Although at least one study found no problem with hand assembly of these cassettes [Puskar et al. 1991], NIOSH and others have occasionally observed, after sampling black or colored dusts, streaks of dust on the filter's compression seal region or an incomplete compression mark, indicating aerosol leakage bypassing the filter. An airtight seal in these cassettes is achieved by compression of two plastic parts that must be parallel and joined with the proper force. If this seal is not compressed with sufficient force, the vacuum behind the filter may pull the filter from the seal, especially at high flow rates. Too high a compression force results in cracking the cassette or cutting the filter, also producing leakage.

Prudence dictates that a check of cassette integrity and the seal area on each filter should be made after sampling to ensure that the cassette was properly assembled; otherwise, the sample may underestimate the actual exposure.

At least two approaches to eliminating the sealing problem with press-fitted cassettes have been taken. One was to assemble the cassette using a press. Pressing the cassette together by hand often produces misalignment of the cassette parts, resulting in bypass leakage. Frazee and Tironi [1987] designed a mechanical press that held the two cassette pieces in proper alignment, while applying just enough pressure to effect a seal, but not so much as to cause cracking of the plastic. This press was designed to allow motion of the cassette pieces to compress to a certain distance. A commercial pneumatic press (Accu-Press^{TM}, Omega Specialty, Chelmsford, MA) used a selected pressure to compress the cassette components. For additional information on bypass leakage and bypass leak test procedures see [Baron 2002]. The second approach was to redesign the cassette to provide a more positive filter seal [Van den Heever 1994]. In a well-designed sampler, opening the seal should not cause tearing and loss of the filter or collection medium during removal from the sampler.

The 37-mm closed-face cassette is usually sealed with tape or shrink bands around the outside. There is a common misconception that this seal prevents bypass leakage in the cassette. These external seals primarily cover the joint between the cassette components to prevent deposited particles on the external surface of the cassette from contaminating the sample during filter removal. The tape or shrink band also aids in holding the cassette together and preventing external air leakage. However, Puskar, et al. [1991] found that even by using this precaution, a significant amount of dust was found downstream of the filter. The authors hypothesized that this dust was deposited during filter removal.

Three other commercial samplers, the IOM (Figure 2B), the CIS, and the coal mine dust sampler (MSA, Inc., Pittsburgh, PA) use a cartridge to hold the filter. For the first two samplers, an external threaded cover applies pressure to the cartridge to ensure a good seal around the filter. This prevents twisting at the filter surface while creating positive, even contact around the filter edge.

5 Electrostatic losses

Most aerosol particles generated in workplaces have electrostatic charge levels considerably higher than the steady-state or equilibrium charge level [Johnston et al. 1985]. The equilibrium charge (Boltzmann equilibrium) levels are usually achieved after particles are suspended in the ambient atmosphere for approximately an hour in the presence of naturally occurring atmospheric ions of positive and negative polarity. When freshly generated particles are sampled in the presence of an electric field, as when a sampler walls are highly charged [Baron and Deye 1990b], the particle trajectories can be modified to such an extent that the particles are inefficiently sampled. No electrostatically induced particle motion occurs when either the particle charge or electric field during sampling is zero. When both the sampler walls and particles are both highly charged, external force on the particles from electrical field is much greater than that caused by gravity, inertia, diffusion or other mechanisms.

Samplers can achieve a high charge level when they are electrically insulated from ground and are triboelectrically charged (i.e., by contacting or rubbing against other surfaces); this sampler charging, as well as particle charging, tends to occur more frequently at low (<20% RH) humidity levels. Certain plastic materials, such as polycarbonate, polytetrafluoroethylene, polyvinyl chloride (PVC), and polystyrene readily retain high charge levels; others, such as Tygon® or conductive silicone rubber tubing retain relatively little charge [Liu et al. 1985]. The PVC/polystyrene copolymer used in the 37-mm closed-face cassette is an excellent electrical insulator and can retain high charge levels on its surface. These charges can be incorporated in the bulk plastic during manufacture or accumulated on the surface by handling or contact with other objects; the charge levels and polarity are highly localized and variable. Such samplers can exhibit particle losses to the internal walls of the cassette and negative sampling biases [Baron and Deye 1990a]. Non-conductive plastic asbestos samplers were shown to produce large negative biases and variable results [Baron and Deye 1990a,b; Baron et al. 1994].

Conductive samplers have demonstrably lower losses when sampling charged particles. Metal samplers obviously have high conductivity. Samples collected using nylon cyclones were shown to exhibit higher variability [Almich and Carson 1974; Briant and Moss 1984] and negative biases [Briant and Moss 1984] when sampling charged dusts. However, the degree of conductivity required is not high; as long as charges can move over the sampler surface and reach equilibrium in seconds, the effect of charges transferred to the sampler is likely to be minimized. Materials with this low level conductivity (surface resistivity <108 ohms/square) are often termed “static-dissipative.” Graphite-loaded plastics were developed that have adequate conductivity to distribute charges over the surface of the cassette (e.g., the 25-mm asbestos sampler). A simple test to ensure adequate conductivity of these samplers can be performed by attaching a good quality multimeter at any two points on the sampler surface. Resistance readings in the range of tens of megohms or less indicate sufficient conductivity for sampling purposes.

Some metals are coated with a thin, non-conductive layer, e.g., anodized aluminum. These coatings may retain a surface charge, but this charge will induce an opposite charge in the conductive layer beneath the surface, effectively canceling out the field produced by the surface charge. Recent measurements at NIOSH using a non-contacting electrostatic voltmeter (Model 300, Trek Inc., Medina NY) indicated that no significant external field (<50 volts) could be produced near an anodized surface by rubbing the surface with various plastics or other materials. Plastic or cellulose-based materials rubbed in a similar manner produced electrostatic potentials measured in the hundreds to thousands of volts. Thus, metals with a thin, insulating surface layer are not likely to produce significant external fields that would affect aerosol sampling.

Digestible cassette inserts or capsules, consisting of a static-dissipative plastic dome directly sealed to a filter, have been developed for metals analysis that substantially reduce the error associated with wall losses [Ashley et al. 2013].

A further electrostatic problem not specifically associated with the cassette is the use of filters made of highly nonconductive materials, such as PVC, polytetrafluoroethylene, or polycarbonate. In addition to having desirable chemical properties, these filters have the advantage of not absorbing water from atmosphere, leading to improved weight stability [Lowrey and Tillery 1979; Bowman et al. 1984; NIOSH 1994]. However, these filters can retain a high electrostatic charge level, resulting in non-uniform particle deposition and even repulsion of particles from the filter surface. Such filters are also more difficult to handle during weighing because of charge effects. Even filters that are normally more conductive, such as cellulose-based filters, can become non-conductive and exhibit non-uniform particle deposition and particle losses at very low humidity levels (<10% RH) [Chen and Baron 1995].

A treatment was developed to make filters more conductive without significantly affecting weighing accuracy or moisture absorption [Mark 1974]. In one study, it was found that applying this treatment to the filter decreased particle losses from 14% to 2% [Blackford et al. 1985]. Anti-static sprays are available that leave a temporary static-dissipative coating on surfaces.

6 Sampler deposition uniformity

Some analytical methods require that sampled particles be deposited uniformly on the filter surface. For instance, asbestos fiber analysis by microscopy requires uniform deposition of fibers on the filter for accurate results. Direct silica analysis of collected filter samples also is improved with uniform particle deposition. Classifiers using inertial or gravitational forces tend to stratify the aerosol stream. A small, high velocity inlet in a sampler, such as the 4-mm opening in the 37-mm closed-face cassette, can also result in the larger particles being deposited in a small central area on the filter. Even sampling at high flow rates through more open inlets can cause a non-uniform deposit [Feigley et al. 1992]. This results in particle deposits that vary in uniformity as a function of particle size. Such deposition patterns are visible when sampling colored particles [Sass-Kortsak et al. 1993]. Open-pore foam classifiers may improve the uniformity of particle deposits on the filter, but have not been thoroughly evaluated [Aitken et al. 1993; Vincent et al. 1993]. Careful design of classifiers to ensure mixing of the aerosol prior to deposit on the filter may result in adequate uniformity [Fang and Lippmann 1995]. Even inhalable samplers or samplers that have no classifier may be prone to non-uniform deposits under certain conditions of sampler orientation relative to gravitational settling, orientation relative to external winds, or when sampling charged particles [Baron and Deye 1990b; Liu et al. 1985; Baron et al. 1994; Chen and Baron 1995]. Flaring the inlet of such a sampler, as in the commercial “bell-mouth cowl,” (Figure 2F, Envirometrics, Charleston, SC), is one approach to improving sample uniformity under anisokinetic conditions [Feigley et al. 1992]. In another study, a sampler having an inlet screen (button sampler, Figure 2D, SKC, Inc. Eighty Four, PA) exhibited improved filter deposit uniformity when compared to a closed-face cassette [Hauck et al. 1996].

The filters in some samplers require support to prevent tearing or distortion of the filter. The support device may cause occlusion of parts of the filter surface, resulting in non-uniform particle deposits [Hook et al. 1983].

On occasion, it was observed that poorly-sized tubing connectors protruded into the 37-mm cassette and touched the filter surface. This caused all the airflow to pass through the filter adjacent to the small area of the connector opening. When undetected, this caused low sampling efficiency and pump failure because of the high pressure drop.

7 Sampler wall losses

Particle deposits on internal surfaces (i.e., wall losses) of the 37-mm closed-face cassette for several hundred field measurements were found to be large and highly variable (2 - 100% of dust collected in the cassette) [Demange et al. 1990]. Another study found only 22% of the dust on the filter, 65% on the upstream portion of the cassette, and 22% downstream of the filter [Puskar et al. 1991]. In a study of an in-line cassette of similar shape, it was found that the internal wall deposition of particles could be largely eliminated by: (a) making the cassette conductive, (b) creating an aerodynamically smooth surface having no corners for eddies to form, and (c) decreasing the diameter of the filtration area so that dust does not deposit on the filter adjacent to the upstream walls of the cassette [Blackford et al. 1985]. By incorporating these three corrective measures, the wall losses in the latter cassette were reduced from 25-30% to 5%. These losses appear to be caused by a combination of electrostatic, inertial, gravitational and diffusion mechanisms.

Another solution to the problem of not capturing 100% of the sampled particles on the filter is to use an internal capsule sealed to the filter. All the particles collected in the combined filter capsule are analyzed. The air stream entering the cassette is surrounded by the cartridge and any deposition on the walls of the capsule is retained for analysis. The IOM sampler for inhalable dust uses this approach by having a cartridge form the inlet of the sampler (Figure 2B). This approach also has been used in the in-line cassette of the coal mine dust personal sampling unit (MSA, Pittsburgh, PA) where an aluminum foil cover is crimped onto the filter. A similar cartridge was designed for the 37-mm closed-face cassette in measurements of pharmaceutical dust [Puskar et al. 1992]. Capsules made of "static dissipative" plastic for gravimetric analysis and capsules composed of cellulosic media for elemental analysis are commercially available (Accu-Cap^{TM}, Omega Specialty Instruments, Chelmsford, MA; Woodchek^{TM}, MSA Inc. Pittsburgh, PA). It is important that capsule material be compatible with the analytical method. For instance, the plastic material used in the first version of the IOM sampler cartridge (Figure 2B, SKC, Eighty Four, PA) was found to absorb milligrams of water over periods of days, making the accuracy of gravimetric measurements problematic [Smith et al. 1997; Li and Lundgren 1999; Lidén and Bergman 2001]. Demange et al. [2002] more recently demonstrated significantly improved agreement between inhalable sampling using the IOM sampler and the 37-mm cassette by including all deposits inside the cassette. This suggests that the accuracy and precision of the 37-mm cassette can be improved by including internal sampler deposits by wiping or washing, or by using an internal capsules [Ashley and Harper 2013; Harper and Ashley 2013; Andrews et al. 2016].

8 Collection media and analytical issues

Interaction of particulate filter with the sampled aerosol and the flow can lead to certain measurement errors, which are sometimes referred to as filter artifacts. These artifacts can include adsorption of gases and vapors from the air stream, the adsorption or desorption of moisture by the filter media, evaporation of volatile or semi-volatile organic matter from the filter media, and particle bounce from the filter media. All these factors can contribute to the measurement bias.

The filter medium should be compatible with the analytical method. Some analytical methods require specific filter media or properties. For instance, atomic absorption and inductively coupled plasma analyses typically require complete ashing of the filter material; organic compound analyses require that no reaction or adsorption of the compounds occur at the filter surface. Several studies have dealt with gravimetric stability of different filter types and recommended specific procedures [Lowrey and Tillery 1979; Bowman et al. 1984; NIOSH 1994; ASTM 2000; Chow 1995; Raynor et al. 2011]. Generally, plastic materials that do not absorb water (polycarbonate, polyvinyl chloride, polytetrafluoroethylene) are more weight stable than natural cellulose-based materials; uncoated glass fiber filters also may absorb water [Lowrey and Tillery 1979; Bowman et al. 1984; NIOSH 1994; ASTM 2000; Chow 1995].

Controlled environmental conditions in the weighing room, where temperature and humidity are strictly controlled, are essential to reduce measurement bias in gravimetric analysis. In a controlled study Tsai et al. [2012] have shown that the mass of MCE membrane filters was less stable than that of the glass fiber filters in both controlled and uncontrolled environmental conditions. Also, they found that under uncontrolled conditions (where humidity and temperatures were not controlled), glass fiber filter mass was much less stable than that of PTFE and PVC membrane filters. MCE and glass fiber filters demonstrated significantly better stability under controlled conditions; whereas the PVC and especially the PTFE filters were found to be extremely stable in both controlled and uncontrolled conditions [Raynor et al., 2011]. Other non-aqueous vapors can also adsorb to the filter media or previously collected particulate deposits. However, these artifacts are typically important only for semi-volatile organic compounds.

It should be noted that weight stable materials also tend to be more highly charged, resulting in more charged particle repulsion and deposit non-uniformity. When a plastic (Tyvek®) backup pad is crimped into a cartridge together with a filter, the weight stability of the cartridge may suffer [Kogut et al. 1999]. To improve the weight stability of coal mine dust sampler cartridges, stainless steel backup pads have been used by MSHA. The IOM sampler can be purchased with either a plastic or a stainless steel cartridge. The plastic cartridge has been shown to exhibit poor weight stability and should not be used for gravimetric analysis [Smith et al. 1997; Lidén and Bergmann 2001].

Lawless and Rodes investigated the use of modern electronic balances to determine factors affecting the accuracy of gravimetric measurements and found that balance stability, balance leveling, vibration and thermal drafts, electrostatic charge reduction, positioning of the filter in the balance so that the filter did not hang over the edge of the pan, and temperature and humidity control were all important in achieving accurate results [Lawless and Rodes 2001].

Although not strictly a problem with the collection medium, the sampler construction material should not outgas vapors that can condense on the collection medium and affect the analysis. Early (circa 1970) versions of the closed-face cassette were made of a plastic called “tenite,” which resulted in weight gain of the filter over time. This currently does not appear to be a problem.

Impactors have been used as samplers and, especially with cascade impactors, the deposits on the impaction stages are measured. Particle bounce from the collection substrates on the impaction plates can be severe, especially for large solid particles impacting onto a smooth metal plate [Marple and Olsen 2011]. Bounce can also be significant for highly nonspherical, low density particles, as was shown recently for nanotubes agglomerates [Birch et al. 2011; Maynard et al. 2004; Baron et al. 2008]. Several modifications to the collection substrate are available to improve collection efficiency of each stage. These modifications should be compatible with the analytical method. Oil can be placed on the collection substrate that wicks up over collected particles and continually provides an oiled surface. To avoid the interference or contamination of particles by oil, the following scheme has been used in recent studies: a pair of cascade impactors was prepared for sampling at a given location. Oiled filters were used on every other stage of each impactor. One of the impactors was loaded with oiled filters on Stages A and C, while Stages B and D were uncoated and used to sample particles onto the substrates. The second impactor contained oiled filters on Stages B and D, while Stages A and C were used for particle sampling. This approach provided data for all four stages (plus after filter) and minimized bounce to the adjacent lower stage [Baron et al. 2008; Birch et al. 2011]. A filter or sintered metal can be used to provide a reservoir for this oil. For gravimetric analysis, this oil must have a low vapor pressure and not migrate off the collection substrate. Alternatively, grease can be used, but after the surface is coated with collected particles, additional particles are more likely to bounce. Filters have also been used as substrates and provide a convenient substrate that is somewhat better than a smooth metal surface. Selection and use of an impactor is a complex issue and has been described in reviews [Lodge and Chan 1986; Marple et al. 2001]. Accurate analysis of cascade impactor data can also be difficult and simple regression analysis of the data may not provide the best answer [Marple et al. 2001; Kandlikar and Ramachandran 1999; Cooper 2001].

9 Sampler field comparisons

Direct field comparisons of various samplers are frequently reported in the literature. Because of the typical high variability of aerosol concentrations and size distributions in workplaces, it is difficult to use these situations for accurate assessment of sampler performance. However, field studies are important to verify the overall performance of a sampler and to indicate specific sampler issues. The problems with samplers as discussed above can be highlighted with some examples observed in field studies.

a. Sampler bias affected by internal deposits

A study of wood dust sampling comparing collocated free-standing samplers indicated that an MSA cassette (having an aluminum cartridge crimped onto the filter) used as a sampler gave two times better precision and collected 2.6 to 3.5 times more dust than the standard 37-mm closed-face cassette [NACSI 1992]. Both these samplers have the same size and shape of inlet. The same study showed that the IOM sampler collected 1.3 times more dust than the MSA cassette, indicating that the particle size, inlet shape and inlet orientation are important factors in inhalable sampling. Among a number of inhalable samplers in current use around the world, the IOM sampler appears to agree the best with the inhalable dust sampling convention [Kenny et al. 1997; Bartley 1998]. Several studies have shown that the IOM sampler collects anywhere from slightly more to 3.5 times more dust than the closed-face cassette [Vaughan et al. 1990; Burdorf et al. 1994; Notø et al. 1996; Perrault et al. 1996; Wilsey et al. 1996]. However, Demange et al. showed that for several work sites with relatively small MMAD (about 15 µm diameter), measurements from 37-mm cassettes agreed well with the IOM results if the deposits on the internal surfaces of the cassette were added to the filter analyte [Demange et al. 2002; Harper and Demange 2007]. Measurements with the 37-mm cassette are not expected to agree as well with the IOM when the particle sizes are much larger because of differences in aspiration efficiency. However, by including all aspirated material, i.e., all material entering the 37-mm cassette inlet, in the analysis, agreement with the inhalable convention can be improved.

b. Sampler precision affected by internal deposits

The issue of measurement bias from internal wall deposits in the sampler has gained increasing recognition over the past few decades [Ashley and Harper 2013]. Though it is now widely recognized that the wall deposits must be included in the analysis, many published methods have not been modified. OSHA currently recommends including wall deposits.

In a field study of lead dust, it was found that the measurements from a closed-face 37-mm cassette gave a coefficient of variation (CV) of 1.0 and 0.33 when sampling at 2 L/min and 10 L/min, respectively, while the button sampler gave a CV of 0.10 under the same conditions [Hauck et al. 1996]. The button sampler has few internal surfaces for wall deposition, suggesting that elimination of this type of loss would improve the precision of the 37-mm cassette. Demange et al. [2002] found improved precision for the 37-mm cassette data when the wall deposits were added to the analyte.

In spite of some of its drawbacks, the 37-mm cassette is likely to be used for some time. It appears that from the standpoint of improving agreement with the inhalable convention and improving precision, the inhalable sampler wall losses should be minimized through sampler design (or through use of a cartridge such as the AccuCap or in the MSA coal mine cassette) or the wall deposits should be included in the analysis.

10 Conclusions

Clearly, when proper features are incorporated in the sampler design, significant improvements in bias and precision can be achieved for some currently used aerosol samplers. Several recommendations regarding the application of these samplers are listed:

- Classifiers used to select respirable, thoracic, or other fractions should be evaluated based on bias maps obtained from experimental data and combined with particle size distributions from workplace measurements to evaluate their applicability.

- Further research and development is needed to improve sampler design to better match ACGIH/ISO conventions and reduce inter-laboratory variability in conducting aerosol sampling. It is important to report the sampler and flow rate used to allow evaluation of potential biases due to sampling. It is also important to account for wall losses to reduce overall bias and allow better comparison across different samplers and ISO standards.

- The filter cassette and fittings should be air-tight and have no bypass leakage. A pneumatic or mechanical press should be used to assemble the cassette and a leak test should be used to establish appropriate pressure and proper assembly procedures. See [Baron 2002]. The verification code for this document is 499785

- The sampler should be made of conductive or static-dissipative materials.

- Internal deposits in sampling cassettes should be included in the analysis. One approach to improving the closed-face cassette measurements is to use an internal digestible cassette insert or cartridge that collects all the sampled dust entering the cassette. The cartridge must be compatible with the analytical method. Another approach is to wipe or wash the internal surfaces of the cassette and add this material to the filter analyte.

Disclaimer

11 References

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