

with a filter.<sup>16</sup> In addition, it would seem logical to use a conductive cassette rather than the clear polystyrene cassette. Conductive cassettes are available in several sizes.

## DEPOSITIONAL PATTERNS AND FIBER COUNTING

A number of studies of depositional patterns of asbestos fibers on filters have been performed, and the overwhelming conclusion is that patterns often are not uniform. The methods for analyzing asbestos (see, e.g., NIOSH 7400) require a maximum of 100 microscopic fields to be examined and the fibers counted. The standard field has an area of 0.00785 mm<sup>2</sup>, and the filter surface area is nominally 385 mm<sup>2</sup>. Therefore, only 0.2% of the filter surface is examined. For this reason, sampling bias in the selection of the fields can be important.

Baron and Deye (1990a) showed that electrostatic charge can have a significant effect on deposition. They recommend that if few particles are found along the edges of the filters, then the fields should be selected from the middle of the filter. This is sometimes difficult when the filter is cut radially.

Baron et al. (1994) have examined deposition patterns at various flow rates (2-10 L/min) with the cassette oriented at various angles relative to a moderate flow field of 0.14 m/sec (28 ft/min). The patterns indicate clearly that at higher sampling rates, non-uniform distributions prevail. Higher rates are, however, warranted so as to avoid complete loss of fibers to wall deposition.

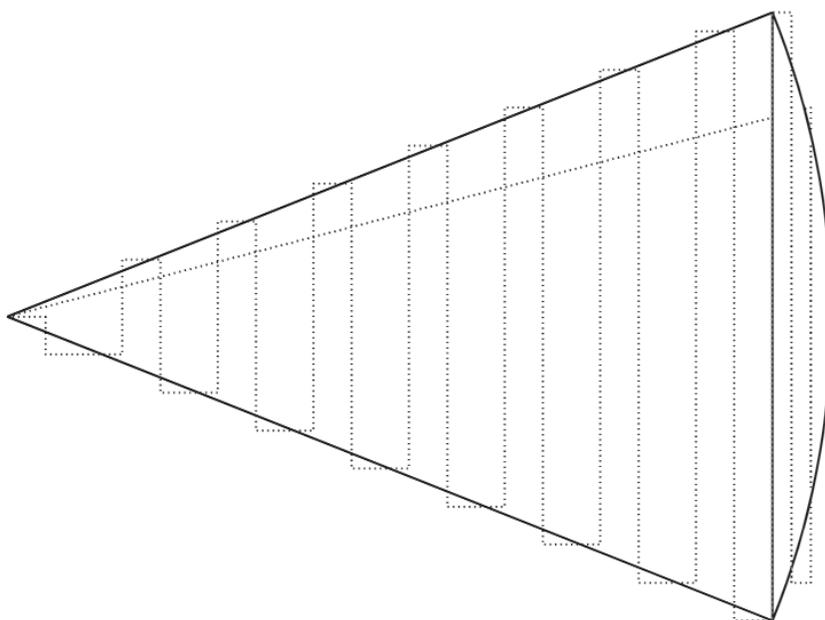
A number of studies by Feigley and coworkers (Hook et al., 1983; Jenkins et al., 1992; Feigley et al., 1992) confirm higher densities near the middle and a number of other irregular patterns. In the most recent study (Feigley et al., 1992), nine cassette types were examined. Interestingly, three gave effective filter surface areas of 420, 404, and 401 mm<sup>2</sup> rather than the assumed value of 385 mm<sup>2</sup>. Failure to account for this would lead to underestimates of the concentrations. At pump flow rates of 9.5 L/min,<sup>17</sup> they found a clear trend of high to low fiber densities from the center to the edge of the filter. The effects were minimized for a cassette with a bell-shaped entry.<sup>18</sup> They concluded that if the effects are caused by electrostatic losses to the walls, then counting fields in the middle of the filter would be reasonable. However, if the patterns are owed to air streamlines caused by the shape of the cassette entry, then counting only fields in the middle would give positively biased results. If the analyst is not sure what is occurring, an approach that randomly selects fields from across the entire filter (or wedge) is probably most pragmatic. One such approach is shown in Figure 24-10.

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<sup>16</sup> See earlier comment; this is apparently available from MSA.

<sup>17</sup> Flow rates of this magnitude are commonly used in the United States for stationary measurements performed to release for occupation a previously abated area, usually an office area where asbestos has been removed.

<sup>18</sup> Available from Envirometrics, Inc., Charleston, SC, USA, no website.



**Figure 24-10.** A diagram of a wedge cut from a filter and a counting pattern that can be used to cover the entire filter. The analyst has to become accustomed to flicking the wrist so that about 100 movements cover the entire wedge. Fields should be randomly selected.

### WEIGHT INSTABILITY OF THE IOM AND CIS INTERNAL CASSETTE

The first cassettes in the IOM samplers were stainless steel and later were also made of plastic to cut costs. This led to the discovery that the plastic cassettes were extremely subject to weight fluctuation caused by humidity (Smith et al., 1998). Liden and Bergman (2001) observed that the carbon black mixed into the plastic from both the SKC IOM and the CIS (conical inhalable sampler) to make them electrostatically conductive is the reason for the water absorption; however, they also observed that not all carbons have the same absorptive properties. A substitute conducting plastic was found to have 30 times less absorption of water.

The bottom line is that the plastic cassettes will absorb water and so careful pre- and post-handling of the cassettes is necessary before weighing, otherwise the cassette-absorbed water may contribute 1-2 mg of weight. This also means that, even with careful procedures, the differential water gain from one cassette to another will contribute to the variability of the method, which contributes to the determination of the smallest mass that can be detected (the LOD, see ASTM, 2006 for methodologies related to filter/cassette weighing) and the smallest mass that can be quantified (the LOQ, see next chapter). Paik and Vincent (2002) found these values to be 0.19 and 0.65, respectively. This is considerably larger than the LOD NIOSH reports for the weighing of filters only from 37 mm cassettes, 0.03 mg (NIOSH, 1994). In 2004, SKC changed the plastic material to address static and water vapor issues and the weight

stability concerns raised by Paik and Vincent (2002). The cassette cannot be desiccated, only equilibrated at room humidity (personal communication, D. Dietrich, SKC).

## RESPIRABLE PARTICLE MASS<sup>19</sup>

### Cyclones

Cyclones are devices that separate particles according to their angular momentum. The cyclone imparts an angular acceleration<sup>20</sup> to the particle due to its cyclonic shape (see Figure 24-11). The momentum of the particle is the product of its mass and instantaneous velocity. Particles with higher masses will be intercepted by the sides of the cyclone, fall to the bottom, and not be collected on the filter. If the flow rate through the cyclone increases, the momentum of all of the particles increases; thus a larger portion is not collected on the filter. This is depicted in Figure 24-12. Note also that the slopes of the curves in Figure 24-12 are not steep. In other words, the cyclone is not 100% efficient up to some particle size and 0% efficient beyond that particle size. However, some cyclones give steeper curves than others (Saltzman and Hochstrasser, 1983; Saltzman, 1984) and all cyclones have steeper efficiency curves than the RPM curve (Trakumas and Hall, 2003 and see Figure 24-13).

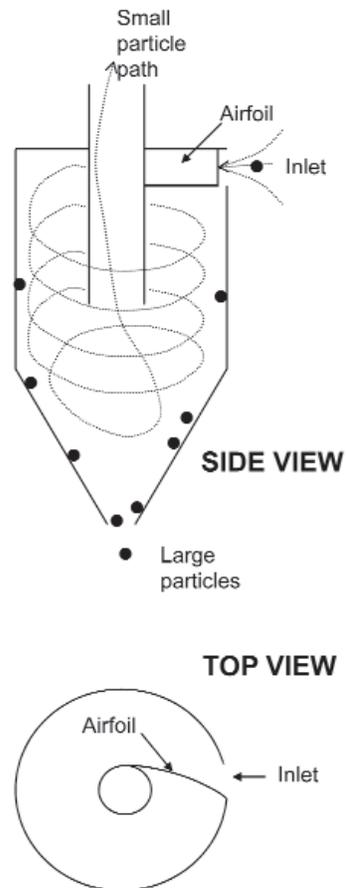
Historically, two cyclones were used to collect *respirable dust*. In the United States, the 10 mm nylon cyclone (see Figure 24-14), also known as the Dorr-Oliver (name of the manufacturer, in Stamford, CT), was used, and is still used by OSHA, at a flow rate of 1.7 L/min to achieve a 3.5  $\mu\text{m}$  cut-point and the ACGIH<sup>®</sup> criteria (see Table 24-6). It is used at a flow rate of 2.0 L/min to achieve the BMRC cut-point of 5  $\mu\text{m}$ . The U.S. Mine Safety and Health Administration (MSHA) still uses the 2.0 L/min flow rate and the correction factor of 1.38, thereby attaining the BMRC criteria, rather than the old ACGIH<sup>®</sup> criteria.

In the United Kingdom, the BMRC criteria were used. Originally, a cyclone was designed to achieve the BMRC criteria (Higgins and Dewell, 1967), which was known as the British Cast Iron Association (BCIRA) or Higgins and Dewell cyclone. Later, the Safety in Mines Personal Equipment for Dust Sampling (SIMPEDS) cyclone was developed as a modified alternative, but over time it has been further modified. Because of the weight of the SIMPEDS (it was brass), a Scandinavian cyclone was developed out of aluminum (Liden, 1993). It was modified several times and is now manufactured by SKC (see Figure 24-15). A more original version of the SIMPEDS is manufactured by BGI (BGI-4).

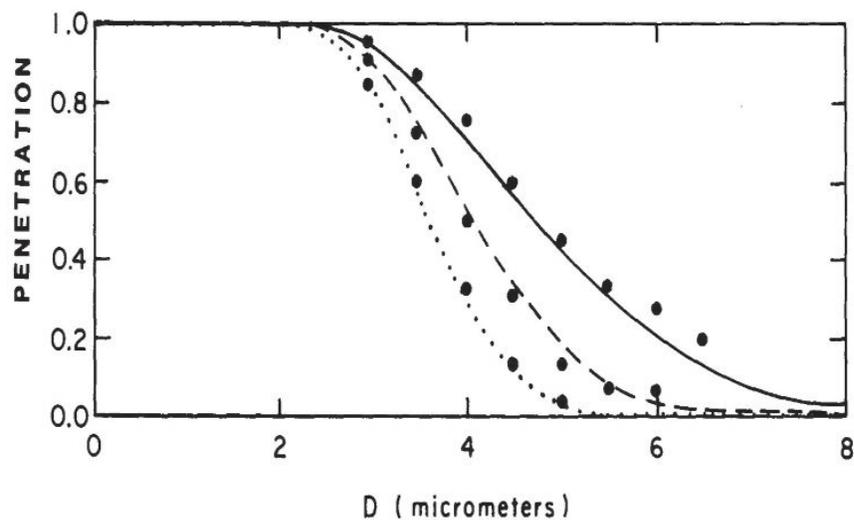
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<sup>19</sup> Again, while this and the other fractions are often referred to as mass fractions, they can just as easily be assayed as number or surface area fractions. The term *mass* refers to the way it is assayed, usually weighed.

<sup>20</sup> Velocity is a vector denoting both speed and direction. Acceleration is a change in speed or direction. Thus, when the particle speed is constant, but the particle has a circular motion, it is accelerating.



**Figure 24-11.** Two views of a cyclone. Entering air is given a cyclonic movement so that large particles are thrown against the cyclone side and fall out. The filter would be downstream from the top exit.



**Figure 24-12.** Modeled cyclone penetration curves and experimental data: solid = 1.4 L/min; dashed = 1.7 L/min; dotted = 2.0 L/min (from Bartley and Breuer, 1982, with permission).

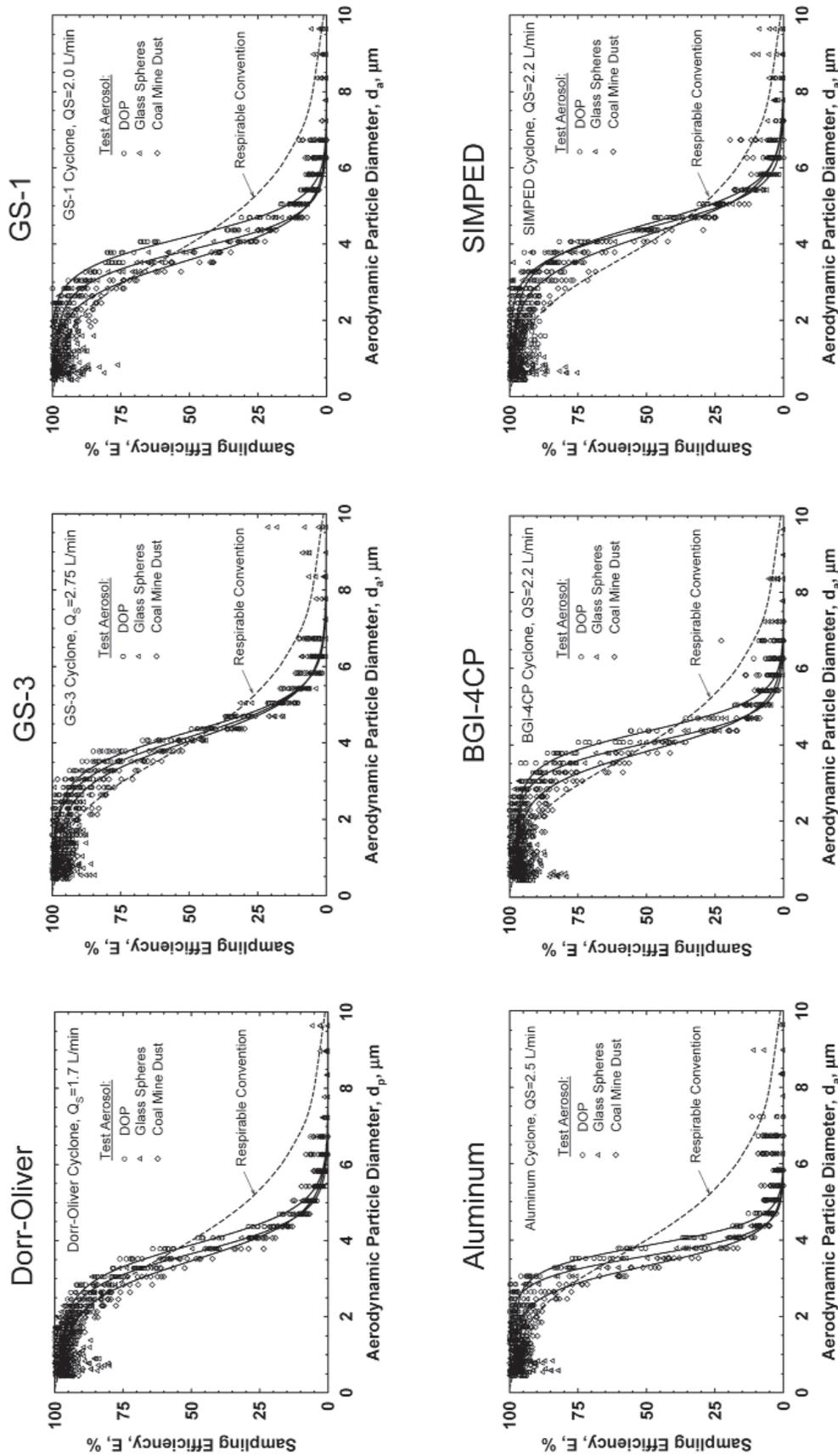
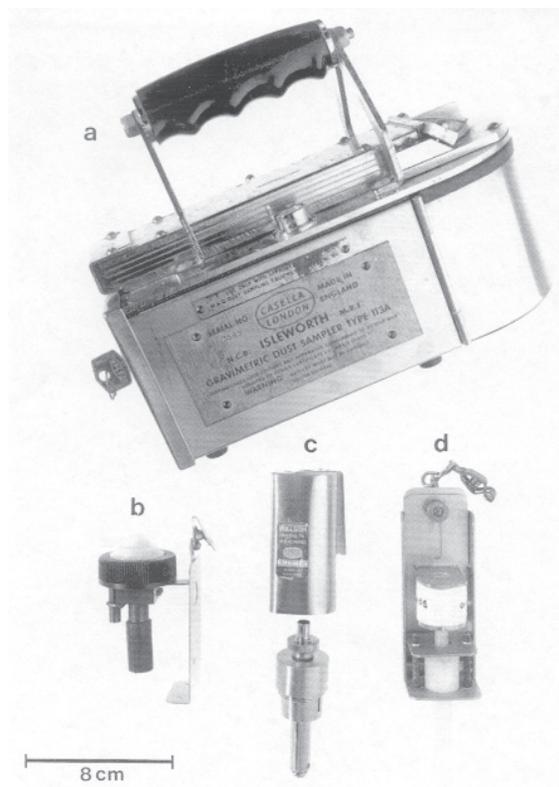


Figure 24-13. Penetration curves for three test aerosols (from Trakumas and Hall, 2003, with permission).



**Figure 24-14.** Historic size-selective aerosol samplers: a) MRE; b) SIMPEDS cyclone; c) BCIRA cyclone; and d) 10 mm nylon cyclone with cassette and holder (from Verma et al., 1992, with permission).



**Figure 24-15.** The SKC aluminum, the 1-hole inlet (simulates the Dorr-Oliver), and the 3-hole inlet cyclones (courtesy SKC, Inc.).

**Table 24-6**  
**Personal respirable size selective samplers (references below<sup>1</sup>)**

Sampler	Other names	Source	Q <sub>4</sub> L/min	Q <sub>3.5</sub> L/min	Filter (mm)
nylon cyclone <sup>2</sup>	Dorr Oliver (D-O)	MSA, Sensidyne	1.7 <sup>3</sup>	1.4 <sup>4</sup>	37
GS-3 cyclone	D-O like, but 3 inlets	SKC	2.75	3.7	25/37
GS-1 cyclone	D-O equivalent, 1 inlet	SKC	2.0	3.0	25/37
SKC aluminum	Modified SIMPEDS	SKC	2.5	2.8	25/37
BGI-4 or CAS-4 cyclone <sup>3</sup>	BCIRA or Higgins Dewell-like, SIMPEDS Casella plastic cyclone	BGI, CasellaCel	2.2		25/37
PEM impactor	Marple-Miller, 1 stage inertial impactor	MSP, SKC	2, 4, or 10 <sup>5</sup>		37
Respirable PPI	Parallel particle impactor	SKC	2		37
Polyurethane foam	Modified IOM, dual fraction	SKC	2		25

<sup>1</sup> MSP, 2003; Gorner et al., 2001; SKC, OSHA (2003), MSHA (2003), BGI (2006), MSA (1998)

<sup>2</sup> non-conducting and required for OSHA/MSHA inspection officers

<sup>3</sup> MSHA uses 2.0 and a correction factor of 1.38 for an equivalent cut-point of 5 µm

<sup>4</sup> OSHA calibrates at 1.7, but allows 1.5–2 for a valid sample

<sup>5</sup> available at 2.5 µm cut only

Considerable research on these cyclones has been prompted by the Universal particle size-selective convention. The research shows that the existing cyclones can be operated to meet the new criteria and, in some cases, that the operating parameters of the past (and present for OSHA) may not have been achieving the old criteria (see Table 24-6 for the nylon cyclone). Bartley and Breuer (1982) calculated that the 10 mm nylon cyclone optimally met the old ACGIH<sup>®</sup> criteria when operated at 1.4 to 1.5 L/min, rather than 1.7 L/min (see also Gorner et al., 2001). In addition, it met the BMRC criteria better if it was operated at 1.2 L/min with a correction factor (*K*) of 0.91, rather than at 2.0 L/min with *K* = 1.38 as used by MSHA. Liden and Kenny (1993) found that the nylon cyclone was optimized for the BMRC criteria at a flow of 1.5 L/min, no correction factor. Ironically, it has been shown that the nylon cyclone meets the new, universal criteria when operated at a flow rate of 1.6 to 1.8 L/min (Liden and Kenny, 1993). While most U.S. hygienists (including OSHA, but with the exception of MSHA) have been operating at 1.7 L/min, they have been unwittingly using the new particle size-selective criteria all along. Optimization of the performance of the SIMPEDS and the

SKC<sup>21</sup> cyclones has also been calculated (Liden, 1993; Liden and Kenny, 1993).

## OPTIMIZATION CRITERIA

Optimizing the performance of a cyclone is not a straightforward task. In past studies, many flow rates have been examined, because the shape of the penetration curve will change a bit with each flow rate. Generally, however, we cannot adjust the flow rate so that the universal RPM criteria and the cyclone penetration curves exactly coincide, and of course even if we could, Figure 24-13 shows that the curves would change for different aerosols. The work summarized in Figure 24-13 shows the variability in efficiency for three different aerosols. This is simply the nature of cyclones.

How then do we adjust  $Q$  so that the performance of the cyclone is most like the criteria? We could calculate the root mean square<sup>22</sup> difference for various points along the two curves; but as we ultimately are interested in the total weight of particles that is selected by the cyclone, differences at the right-hand tail (heavier particles) are more important than those at the left. Therefore, aerosol size distributions that are *large* will be sampled with a different bias from that of an aerosol that is distributed at the *small* end of the spectrum. Bartley and Breuer (1982) proposed that the cyclone be optimized, by finding the flow rate that gave the smallest average bias in the weight of particles collected when compared to the appropriate particle size-selective criteria. They proposed that the average bias could be calculated across a range of aerosol distributions representative of all industry, or that preferential weighing could be given to a *region* of dust distributions, such as those found in the coal industry.

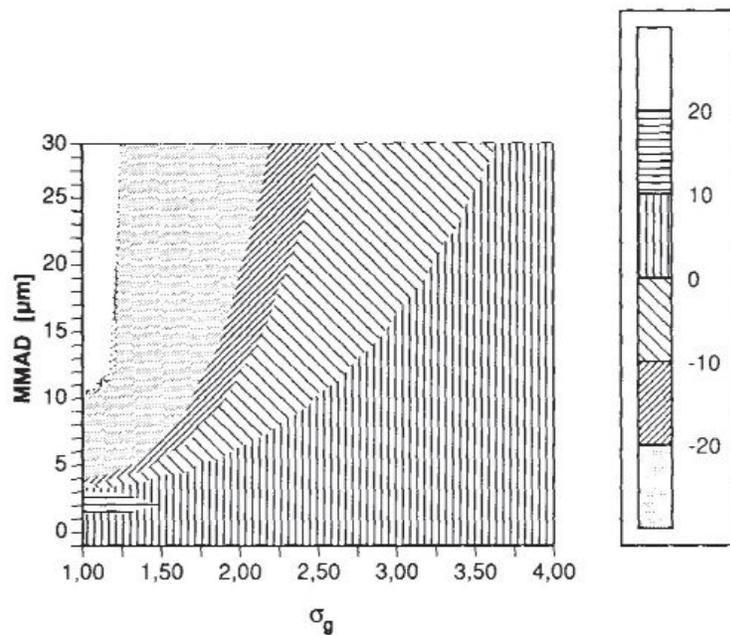
To this end, Liden and Kenny (1993) recommended three testing methods that comprised up to 153 different aerosol distributions or combinations of mass median aerodynamic diameters (MMAD) and geometric standard deviations (GSD) including one approach consisting of 34 distributions based on the actual workplace measurements of Hinds and Bellin (1988). As these proposed evaluations were across a large number of distributions, a laboratory study is not feasible. Instead, Bartley and Breuer (1982) proposed the use of mathematical models that yield penetration curves for any flow rate that are based on empirical penetration data derived at one flow rate for that cyclone. These modeled penetration curves are studied to determine the optimal flow rate for the range of aerosols likely to be encountered.

From the optimal flow rate, one can then find the bias associated with each aerosol size distribution, a bias map. Figure 24-16 shows such an example for the nylon cyclone. Generally, the bias is less than 10%, but in some cases it can be quite high. An important conclusion can

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<sup>21</sup> Since 1986 there have been at least 4 generations of what is now the SKC aluminum cyclone.

<sup>22</sup> The root mean square is similar in concept to the standard deviation. It is an attempt to derive an average difference by neutralizing the effect of the algebraic sign of the individual differences. Thus, each difference is squared, the square differences are averaged, and the square root of the mean is taken.



**Figure 24-16.** Modeled optimization graph for various lognormal aerosol size distributions (based on the original data of Blachman and Lippmann, 1974) showing contours of equal bias (%) for the 10 mm nylon cyclone ( $Q = 1.7$  L/min in calm air and exposed to ferric oxide monodisperse aerosols) relative to the universal RPM convention (from Liden and Kenny, 1993, with permission).

be drawn: the error caused by the use of a non-optimal flow rate may be small compared to the error caused by the aerosol distribution. Thus, a relatively inexpensive way of determining the actual aerosol distribution would be important so that correction factors can be made (from a bias map) for cyclonic sampling. Finally, there have been a number of studies that determined optimum flow rates for cyclones to meet the Universal RPM (see, e.g., Kar and Gautum, 1995; Liden and Gudmundsson, 1996; Tsia and Shih; 1995 as well as other papers cited above); however, in the past decades manufacturers have changed designs, recommended and then retracted the use of correction factors, and changed recommended flow rates. It is, therefore, best to use the flow rate recommended by the manufacturer at the time the device was purchased or inquire directly with the manufacturer where there is a discrepancy between the original and a current recommended flow rate.

## PRECISION AND BIAS

It should be apparent now that particle size-selective samplers are more difficult to evaluate for precision and bias than are other sampling methods such as charcoal tubes. A different bias and precision is possible for each aerosol size distribution. It is difficult to devise an

overall statement of the accuracy (precision and bias) of particle size-selective samplers for comparison with the often cited NIOSH requirement that a method have an accuracy of  $\pm 25\%$  at the 95% confidence level for a single measurement (or, roughly stated, the absolute value of the bias plus two times the *CV* should not exceed 25%). For a review of thinking on the subject, the reader should see Bartley and Doemeny (1986), Kenny and Liden (1989), Liden and Kenny (1992), and Bartley and Fischbach (1993). Approaches for calculating the bias have been proposed, which can be accomplished with mathematical modeling. Precision assessments, however, require replicate sampling and analyses in differing aerosol environments (including varying and representative wind speeds, particle charge, and relative humidity effects). The CEN (2001) has devised an accuracy criterion that takes into account the cyclone bias plus the pump and weighing or analytical error. Apparently, it does not include variability from one sampler to another. A parallel ASTM method does (ASTM, 2001b).

The most comprehensive study of RPM samplers to date examined the bias and accuracy of 15 RPM samplers used throughout the world (Gorner et al., 2001). The samplers were assessed against generated and modeled aerosols of 1 to 25  $\mu\text{m}$  (1  $\mu\text{m}$  steps) MMAD and 2.0 to 3.5 (0.25 steps) GSD for a total of 175 aerosols. A bias criterion of  $< 10\%$  (absolute value) was considered acceptable for each aerosol. The percent of aerosols sampled with less than 10% bias were: Dorr-Oliver (84%), SKC plastic (58%), Casella plastic (68%), and SKC aluminum (86%). As Figure 24-13 shows, often cyclones undersample the larger particles. Thus, for each of these cyclones, biases were greatest for the larger ( $> 10 \mu\text{m}$  MMAD) aerosols. This will cause an underestimation of the concentration for aerosol distributions dominated by larger particle sizes.

Chen et al. (1999) studied 25 replicates of SKC aluminum and 10 mm nylon cyclones for both liquid (dioctyl phthalate) and solid (potassium sodium tartrate) aerosols. Results showed that both cyclones were least precise at aerosol sizes around 5  $\mu\text{m}$  (CVs in the 5 to 10% range) while at smaller particle sizes CVs were typically around 5%. Note that most of the variability was not within variation, i.e., variability caused by repeated measurements on the same cyclone, but rather due to between variation or lack of consistency between specimens of the same cyclone type. As a result, manufacturing quality control is questioned. In addition, cleaning cyclones after each use is extremely important. In terms of bias, as compared to the Universal RPM convention, both cyclones typically gave biases less than  $\pm 5\%$  for aerosols from MMAD 1 to 25  $\mu\text{m}$  and GSD from 1 to 3.5. However, for solid aerosol sizes around 5  $\mu\text{m}$ , biases peaked at  $-(10-15)\%$  for the SKC and  $-10\%$  for the nylon sampler. In both cases, the greatest biases occurred for very uniform particles (GSD  $< 1.5$ ), indicating that cyclones should be rotated between SEGs; as particle size distributions become monodisperse and approach 5  $\mu\text{m}$ , sample sizes should increase; and the hygienist should be aware that, for these same conditions, concentrations are more likely to be underestimated.

The precision of any cyclone will be affected by variations in pump flow rate, highly charged particles (depending on the charge distribution on the cyclone), and relative humidity.

## Pump Fluctuations

Piston and diaphragm sampling pumps do not give smooth flow profiles over short time periods, even with pulsation dampeners. When the chamber empties, the flow is highest; and when the chamber fills, the flow is lowest (see Chapter 20, Figure 20-5). This periodicity is caused by the frequency of the motor as it turns the drive shaft. Because cyclone performance is dependent on flow rate, one might expect that the nature of the periodicity is important. Bartley et al. (1984) showed that the effect on cyclones was generally less than 5%, but could be as high as 22% (Berry, 1992) and when the flow variation was controlled to  $\pm 20\%$  of mean flow, the error was  $< 2\%$ . It is likely that newer *constant-flow pumps* adequately control these fluctuations.

## Electrostatic and Humidity Effects

Cyclones can be non-conductive (10 mm nylon) or conductive (metal or carbon filled plastic). As with cassettes, the charge should be concentrated on the outside surface. One might expect that the most variable results would be obtained when charged nylon cyclones were used to sample charged aerosols, and that conducting cyclones would behave similarly, regardless of the aerosol charge.

Almich and Carson (1974) examined the effect of charge on the performance of nylon cyclones under worst-case ( $RH < 10\%$ ) conditions. They found that nylon cyclones retained more particles (i.e., the filter would undersample) when the particles (coal dust, 4-5  $\mu\text{m}$  MMAD) were charged (67%) than when not (52%). A specially constructed stainless steel cyclone (presumably like the 10 mm nylon) also retained more charged (69%) than uncharged particles (56%). Liden (1993) found that the variation in collecting Carborundum particles, which were thought to be highly charged, was much greater than for foundry dusts. Briant and Moss (1984) also showed that the nylon cyclone allowed less penetration of charged polystyrene latex particles (0.5, 1, 2, and 2.8  $\mu\text{m}$  MMAD), but only if the cyclone was charged. A specially constructed, graphite-filled nylon cyclone was not affected by particle charge.

At low humidity levels, the nylon cyclone undersampled silica relative to the horizontal elutriator (see later discussion) even more than when humidity was high. The effect was not seen in sampling of wood dust (Sass-Kortsak et al., 1993). Chen and Huang (1999) found that the 50% cut-point for the 10 mm nylon cyclone became a 30% cut-point (the entire curve was shifted left) when sampling small particles (CMD, 3.5  $\mu\text{m}$ ; GSD, 1.3) under 10% humidity conditions and that the cyclones became dirty from deposits under these conditions, which was assumed to change their sampling dynamics. The nylon cyclone has sampled less than the SKC aluminum in field studies, possibly due to these effects (Verma et al., 1992; Sass-Kortsak et al., 1993; Groves et al., 1994).

The results indicate that charged particles (1) lead to less precision regardless of the conductive nature of the cyclone and (2) may lead to undersampling, certainly for the nylon cyclone (in the case of conducting cyclones, the cyclone would appear to need to carry a