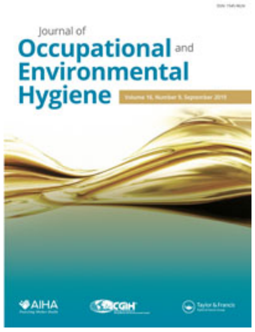

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Evaluation of a 25-mm disposable sampler relative to the inhalable aerosol convention

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ABSTRACT
An ideal inhalable aerosol sampler for occupational exposure monitoring would have a sampling efficiency that perfectly matches the inhalable particulate matter (IPM) criterion. Two common aerosol samplers in use worldwide are the closed-face cassette (CFC) and the Institute of Occupational Medicine (IOM) sampler. However, the CFC is known to under-sample, with near zero sampling efficiency for particles >30 µm, whereas the IOM, considered by many to be the "gold standard" in inhalable samplers, has been shown to over-sample particles >60 µm. A new sampler in development incorporates characteristics of both the CFC and the IOM. Like the CFC, it would be disposable, have a simple design, and is intended to be oriented at a 45° downward angle. Like the IOM, the new sampler has a 15-mm inlet diameter and incorporates a 25-mm filter cassette with a protruding lip. The IOM is oriented at 0° to the horizontal, so it is hypothesized that orienting the new sampler at ~45° downward angle will reduce oversampling of larger particles. In comparison, the CFC's inlet diameter is 4 mm; increasing the size of the inlet should allow the new sampler to have an increased efficiency relative to the CFC for all particles. A unique characteristic of the new sampler is the incorporation of a one-piece capsule-style filter that mimics the IOM's cassette but is made of disposable material. Seven different sizes of alumina particles (mean aerodynamic diameters from 4.9–62.4 µm) were tested (total = 124 samples collected). For each test, six samplers were placed on a manikin located inside a wind tunnel operated at 0.2 m/sec. Results indicated that the new sampler improved on the CFC for smaller particles, providing a larger range for which it matches the IPM criterion, up to 44.3 µm. However, the efficiency was significantly lower in comparison to the IPM criterion for particle sizes above 60 µm. Overall, the new sampler showed promise, but additional modifications may help improve sampling efficiency for larger particles.

KEYWORDS
Aerosol sampler; closed-faced cassette; inhalable particulate matter; IOM sampler; wind tunnel

Introduction

Epidemiological studies have shown a correlation between inhaled particle exposures and adverse health effects.[1] Evidence suggests that being exposed to nonspecific occupational dusts, gases, and fumes increases the risk of chronic respiratory symptoms and decreases the level of pulmonary function.[2] In addition, the American Heart Association (AHA) has concluded that exposure to airborne particulate matter contributes to cardiovascular morbidity and mortality,[3] and others have found that occupational exposures to particles have a possible association to ischemic heart disease.[4] Currently, there is some dispute as to which factor has a greater effect on negative health outcomes (including both respiratory and heart problems): exposure to specific chemical species or simply being exposed to any particulate matter.[5]

Where particulate matter deposits within the human body is determined by the size fraction of the inhaled particles.[6–8] The site of particle deposition, in turn, is strongly associated with health outcomes.[9] The current classification system for exposure to airborne particles is based on size and consists of three main categories or fractions—inhalable, thoracic, and respirable—each one named for the region of the respiratory tract to which the particles can penetrate.[6–8] For example, any airborne particles that can be inhaled through the nose and mouth make up the inhalable fraction; this includes particles that can

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deposit anywhere in the respiratory tract, including the thoracic and respirable subfractions.[6–8] Each fraction is defined by a mathematical function (e.g., Equation [1]) that describes the probability that a particle with a given aerodynamic diameter will penetrate to that region of the respiratory tract.[6–8]

Using a sampler that performs in an equivalent fashion to human inhalability is an ideal goal for workplace exposure assessments.[10] To this end, an inhalable particulate mass (IPM) sampling convention was developed in the early 1980s.[11,12] This convention was ultimately adopted by the American Conference of Governmental Industrial Hygienists (ACGIH®), the European Committee for Standardization (CEN), and the International Organization for Standardization (ISO):

$$\mathrm{IPM} = 0.5\left(1 + e^{-0.06d_{ae}}\right), \quad (1)$$

where d_{ae} is the aerodynamic diameter, in μm, of a particle being sampled.[6–8]

The criterion is meant to approximate human aspiration efficiency based on aerodynamic diameter, and is the current metric against which size-selective aerosol sampler performance is compared.[6–8]

Currently, the closed-face cassette (CFC) sampler is the most widely used aerosol sampler in the U.S.[13] However, the CFC sampler does not meet the IPM criterion for particles larger than 30 µm,[14,15] which is well below the upper limit for the inhalable fraction of 100 µm. In this way, the CFC has historically been misclassified as a "total" dust sampler. Sampling methods promulgated by U.S. agencies such as the National Institute for Occupational Safety and Health (NIOSH) (in the *NIOSH Manual of Analytical Methods (NMAM)*) and the Occupational Safety and Health Administration (OSHA) (in its online manual on air sampling and analysis) both specify the use of the CFC sampler.[16,17]

The CFC utilizes a 4-mm diameter inlet, through which sampled air is drawn in from the atmosphere, passes through a 37- or 25-mm filter, and leaves via a 4-mm outlet where a hose connects the sampler to a pump. The small inlet may limit the aspiration of larger particles, which would still be considered inhalable.(15) The CFC can be used with either two or three cassette pieces, with the three-piece cassette including an additional plastic ring placed between the inlet and outlet sections. A press-fit design, which utilizes the shape of the object and friction between the sections, is used to secure the parts of the CFC together. The design of the CFC is also such that there is space between the inlet and the filter, resulting in particles depositing on the inside walls of the sampler.[18,19] With particles collecting on the walls, there is a potential for the CFC to underrepresent analyte when analysis is performed without inclusion of these deposits.[20,21] Consequently, it has been recommended to include internal CFC sampler wall deposits, along with material collected on the filter, as part of the sample.[22]

Another factor potentially causing the CFC to under-sample particles >30 µm is the inlet orientation with respect to the wind direction. In practice, the CFC hosing is draped over a worker's shoulder and connects to a pump at the waist, with clips holding the pump tubing in place. This places the CFC at approximately a 45° angle down from the horizontal. Witschger et al. suggest samplers with a downward orientation do not create a sufficient flow field to change the direction of rapidly settling particles, that is, the sampling flowrate cannot overcome gravity and pull large particles into the sampler.[23] Cook et al., however, found no evidence that the sampler orientation (45° vs. 0°) resulted in different concentrations in a controlled laboratory study.[24] Kauffer et al. investigated CFCs in the field and found that a 45° downward orientation collected 1.35 times less than a cassette with a forward orientation.[25] Despite these varying results, adjusting inlet orientation may be a viable option for increasing the efficiency of the CFC sampler.

Previous studies have tried to address the modification of a CFC's orientation and inlet size, with some success.[20,26] Computational fluid dynamics (CFD), performed by Anthony et al., demonstrated a modification as simple as cutting a hole in the sampler cap could allow the CFC to perform at the same efficiency as human aspiration for all particle sizes when facing the wind.[26] They suggest that an inlet diameter of 15 mm would increase efficiency, while also limiting the over-sampling of particles larger than the inhalable fraction (>100 µm). Clinkenbeard et al. compared a modified CFC to an Institute of Occupational Medicine (IOM) sampler—the most commonly used sampler worldwide for personal inhalable aerosol sampling.[20] The CFC had been modified to have an enlarged inlet (15 mm diameter), a backing plate, and brass elbow to orient the sampler at 0° to the horizontal. These modifications resulted in the CFC slightly over-sampling in comparison to the IOM.[21] This is likely due to the lack of a protruding lip around the inlet orifice, which is present on the IOM. Results from the CFD study mentioned previously found that this lip does indeed reduce sampling efficiency.[26]

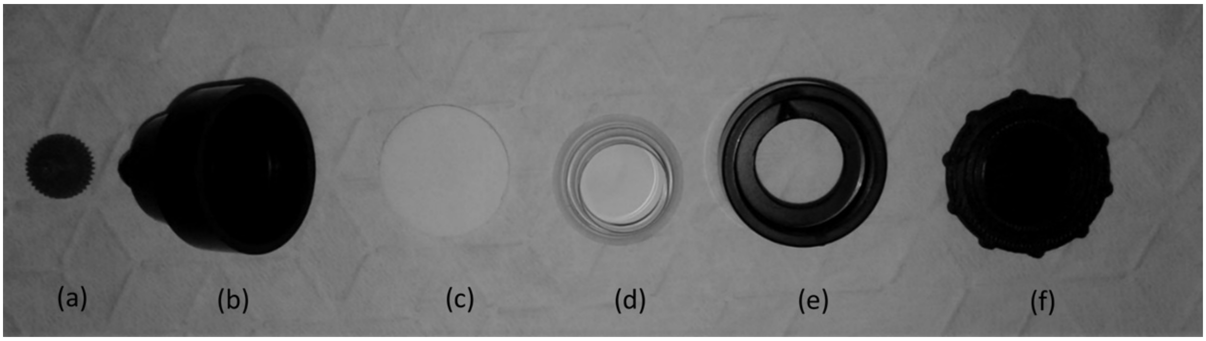

Figure 1. Disassembled parts of the new sampler, from left to right: (a) outlet plug, (b) sampler body, (c) 25-mm diameter backing pad, (d) capsule-filter, (e) 15-mm diameter sampler inlet, and (f) transport cap.

The IOM is considered by many to be the "gold standard" for sampling inhalable particles. Developed in the mid-1980s,[27] the IOM became popular because, in experiments carried out in wind tunnels, it was shown to sample similar to the IPM criterion.[14,28] Worn on the lapel, the IOM is meant to sample close to a worker's breathing zone, thereby allowing for particle collection similar to what a worker would inhale.[29] The IOM is more complex than the CFC, and consists of seven pieces. It has a base that allows it to be oriented 0° relative to horizontal, a total of three O-rings, and an inlet that is screwed on. The inlet (with a protruding lip) is 15 mm in diameter, which is wider than that of the CFC inlet at 4 mm.

One unique aspect of the IOM sampler is the internal cassette that holds a 25-mm filter in place, which must also be analyzed with the filter. The cassette/filter is notable because it has been viewed as difficult to handle,[30,31] although including it in analysis is necessary to provide accurate sampling results.[27] For gravimetric analysis, the capsule is weighed with the filter; thus, unlike non-gravimetric analysis, wiping or washing the sides of the sampler is not needed in order to account for particles deposited on the internal sampler walls.[27] In contrast, the CFC houses a larger, 37-mm filter, although disposable filter-capsules are available to collect internal wall losses in that sampler as well.[32]

A collar clip allows the IOM to be oriented at approximately 0° from the horizontal, such that the inlet is directly facing the oncoming wind. By contrast, the CFC is oriented at a downward angle from the horizontal, typically at least 45°. Sampler efficiency has been shown to change when the IOM is kept horizontal to the ground, but with different orientations (0, 90, 180°) from the wind.[33] The IOM can over-sample up to seven times when facing directly into the wind and under-sample by a factor of 5 when facing 90° to the wind.[34] If changing the orientation of the IOM along the horizontal axis changes its efficiency, it is reasonable to assume that reorienting it downward could also affect efficiency. With increasing particle diameter, the difference between the IOM collection efficiency and the IPM criterion also increases.[33] Thus, similar to the CFC, sampler orientation and particle size both affect the IOM sampling efficiency.

Recent work in inhalable sampler design has sought to mirror the ease of use and familiarity with the CFC, but with better efficiency in regards to meeting the IPM criterion.[31] For this study, a new sampler was designed and investigated, which is hypothesized to better meet the IPM curve based on a combination of design features from both CFC and IOM samplers. The purpose of this article was to assess the sampling efficiency of the new sampler in comparison to the IPM criterion.

Methods

The new sampler for this study was designed with two goals in mind: (1) the sampler should be of a simple design that is easy to use and of low cost, like the CFC; and (2) the sampler should be capable of meeting the IPM criterion.

In order to meet the goal of a simpler design, the new sampler consisted of three main pieces made of static-dissipating plastic and a one-piece filter/capsule (Figure 1). The one-piece 25-mm filter/capsule unit resembles the IOM cassette (i.e., top, bottom, and filter as one piece). Figure 2 shows a side-by-side comparison of the IOM cassette and the capsule-style filter of the new sampler. Replacing the IOM cassette design with a one-piece capsule-style filter design that is of similar shape will likely eliminate some of the perceived difficulty in using the IOM. In addition, the use of the press-fit design imitates the simplicity of

Figure 2. Side-by-side comparison of the IOM cassette (left) and the capsule-style filter of the new sampler (right).

the CFC and eliminates the use of multiple O-rings that are required by the IOM. The capsule-style filter is made from polyvinyl chloride (PVC) and requires a support pad. The one-piece design of the capsule/filter assembly eliminates any need to wipe the inside of the cassette in order to obtain all of the collected material.

In order to meet the goal of improved matching to the IPM criterion, the sampler was designed with a 15-mm inlet (like the IOM), but was oriented at (approximately) a 45° downward angle (like the CFC). It was hypothesized that the larger inlet would improve the sampling efficiency of particles >30 µm relative to the CFC, and that the downward angle would reduce the over-sampling of particles >60 µm at low wind speeds, as has been found to occur with the IOM sampler.[15] With this configuration, it was hypothesized that the newly-designed sampler would incorporate favorable aspects of the CFC and the IOM so as to improve sampling characteristics relative to the IPM criterion. A sampler prototype that met these criteria was manufactured and provided to the researchers by Zefon International (Ocala, FL).

The laboratory tests for this study were conducted in a low-speed aerosol wind tunnel, capable of operating between 0.1 and 0.5 m/sec for simulating indoor air environments (Engineering Laboratory Design, Inc., Lake City, MN). The wind tunnel design, calibration and operating methods have been described previously,[35] and were similarly followed for this study. A life-size manikin torso (Measurement Technology Northwest, Seattle, WA) was placed inside the wind tunnel to hold the samplers. A laboratory coat was placed on the manikin to provide a better replication of air currents around workers' clothing. The manikin rotated 360° two times per minute, reversing direction at the completion of a rotation. Six of the new

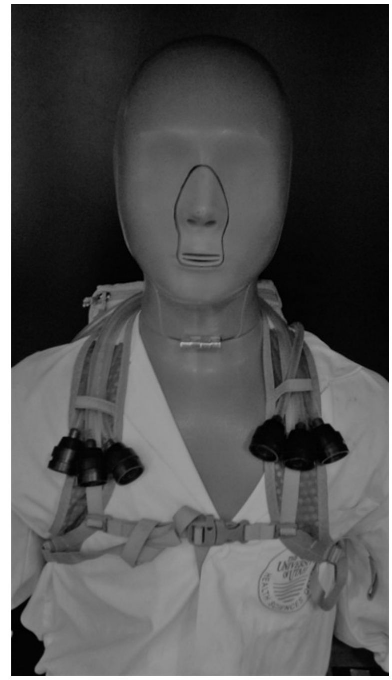

Figure 3. New samplers (n = 6) draped over the manikin shoulders at approximately 45° angle downward from the horizontal.

inhalable samplers were draped over the manikin's shoulders using flexible tubing, three on each side of the neck, at an approximate 45° angle downward from the horizontal (Figure 3). Although the angle of each sampler was not specifically measured, placement was made in a manner that an industrial hygienist might place a sampler on a worker in practice. Each sampler tubing was connected to either a SKC XR5000, SKC 2000, or SKC AirChek TOUCH pump (SKC Inc., Eighty Four, PA) that drew air into the samplers at 2.00 L/min (±5%).

Prior to sampling, each capsule-style filter was analyzed gravimetrically. In lieu of desiccating the capsules, they were acclimatized for several weeks in the laboratory where gravimetric analysis was performed. A semi-microbalance (accuracy: ±0.012 mg) with high voltage neutralizer (Sartorius Cubis MSA, Sartorius Weighing Technology GmbH, Goettingen, Germany) was used for all weighing. Once acclimatized, the pre-weighed capsule was inserted into a sampler, the sampler was assembled, and the cap was put on. Powder-free gloves and tweezers were used when handling samplers and capsule-style filters to avoid contamination before and after sampling. The personal sampling pump flow rates were calibrated with a BIOS DryCal primary flow meter (Bios International Corporation, Butler, NJ) to operate at a flow rate of 2.00 L/min (±5%). All pumps were started simultaneously at the beginning of each sampling event. Samplers whose pump demonstrated a flow rate change of ±5% after each sampling event were excluded from analysis.

Once the manikin was placed inside the wind tunnel with the samplers and personal sampling pumps, aerosols were injected into the wind tunnel via an upstream aerosol dispersion system. The dispersion system uses an air compressor attached to a dust generator (Solid Air Generator 410, TOPAS aerosol generator, Dresden, Germany), and is equipped with a dual tracking system that consists of an injection nozzle mounted on a motor that moves vertically and horizontally.

Seven particle sizes of fused alumina (Duralum, Washington Mills, Niagara Falls, NY) were tested in a series of experiments. The alumina is similar to what has been previously used in aerosol wind tunnels and was characterized in previous studies to have a narrow geometric standard deviation varying from 1.32–1.73.[35,36] Grit sizes were F240, F280, F400, F500, F800, F1200, and F2400, with aerodynamic diameters 60.1, 62.4, 44.3, 32.7, 12.8, 9.5, 4.9 µm, respectively. All particle sizes were tested individually three different times, for a total of 21 sample events (7 grits x 3 sampling events) and N = 126 samples (21 samples x 6 samplers). Speeds in the wind tunnel were representative of indoor air velocities at 0.2 m/sec,[37] and each sampling event lasted for 45 min. This amount of time ensured that concentrations stabilized to a uniform distribution while also collecting enough mass to provide relevant workplace concentrations (between 1-10 mg/m^3). Between runs, a HEPA vacuum was used to clean dust from the wind tunnel and the aerosol generator in order to prevent cross contamination of particles. Sampler holders were thoroughly washed with soap and water between uses. Temperature and humidity were recorded.

After sampling events, care was taken to keep the samplers' inlet pointed in an upward orientation when removing the samplers from the tubing and during transportation from the wind tunnel to the analytical laboratory, in order to avoid loss of collected dust. In the lab, the capsule-style filters were allowed to re-acclimate for at least 12 hr and were post-weighed using the same microbalance as that used for pre-weighing. The pre-sampling weight was subtracted from the post-sampling weight to yield the mass of the collected sample.

In order to calculate the efficiency of the new samplers, a simultaneous measurement of the true air concentration in the wind tunnel was required. To obtain this, for each experiment, two isokinetic samplers were placed approximately 0.75 m upstream of the manikin along the central axis of wind tunnel and with a vertical offset of roughly 15 cm, thereby placing them at a similar height as the new samplers when attached to the manikin. The isokinetic samplers had a 25-mm glass fiber filter secured by an O-ring into a plastic filter holder. A 50-mm long, sharp-edged metal inlet with an 8-mm opening protruded from one side of the filter holder; an outlet on the opposite side of the filter holder was attached to the tubing and sampling pump. The personal sampling pumps of the isokinetic samplers had a flow rate (0.55 L/min) set to match the air velocity in the wind tunnel (i.e., 0.2 m/s). Pumps were calibrated with the same Bios Dry Cal primary flow meter mentioned previously. If any flow rates had changed by ±5% at the end of a sampling event, then that sampler was excluded from analysis.

Gravimetric analysis was used to determine the pre-sampling weight of the isokinetic sampling filters. 2-Propanol was used to remove deposited material from the inside of the metal inlets, which was collected in a pre-weighed glass petri dish and allowed to evaporate for at least 24 hr before the dishes were reweighed post-sampling. That mass was then added to the mass collected on the filters to determine the reference concentration.

Two blank samplers with pre-weighed capsule-style filters were transported from the analytical laboratory to the wind tunnel with the other samplers. Masses for the blanks' capsule-style filters were recorded every time gravimetric measurements were taken. Masses for the blank capsule-style filter did not change between pre- and post-sampling events. Therefore, measurement correction was not necessary.

In order to determine the collection efficiency of the new samplers, dust concentrations for each sampler were averaged and compared to the average reference concentration from the two isokinetic samplers. Then, these sums were multiplied by 100 using the following formula:

$$E_s(\%) = \frac{C_s}{C_o} \times 100, \quad (2)$$

where E_s is the sampling efficiency, c_s is the average concentration measured by the samplers, and c_o is the average reference concentration determined by the two isokinetic samplers. The three sampling events for each particle size were averaged to create a single data point that was compared to the IPM criterion.

Tests for kurtosis and skewness were performed, which confirmed that the data were normally distributed, and a one-sample t-test was then used to compare the new samplers' efficiencies to the IPM criterion at each particle size. Microsoft Excel

Table 1. Averaged efficiency for the new sampler at various aerodynamic diameters, with one-sided t-test results compared to the IPM criterion.

Grit Size	Aerodynamic Diameter (μm)	IPM Criterion (%)	Sampler Efficiency (%)	Standard Deviation	p-Value*
F2400	4.9	86.5	91.0	14.7	0.682
F1200	9.5	77.0	83.4	10.9	0.789
F800	12.8	72.0	55.0	10.6	0.054
F500	32.7	56.2	52.1	14.0	0.331
F400	44.3	52.9	48.3	6.5	0.173
F280	62.4	50.9	19.6	1.5	0.035
F240	60.1	51.1	12.3	4.2	0.016

*p-value <0.05 indicates statistically significant difference

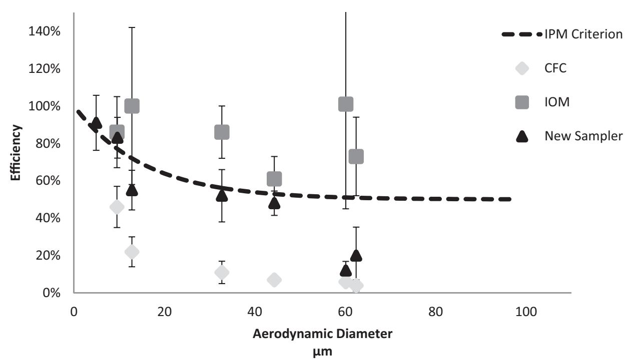

Figure 4. Sampling efficiency of the new sampler for wind velocity of 0.2 m/sec compared to the IPM criterion (dashed line) and to previous experiments with similar methods using the IOM (squares) and CFC (diamonds) at 0.24 m/sec.[15] Error bars represent one standard deviation.

(Microsoft, Redmond, WA) was used to perform the statistical analysis.

Results

A total of 124 samples were obtained across the seven particle sizes. This does not include two samplers (one each for the 32.7 μm and 62.4 μm particle sizes) for which the pumps malfunctioned. For three sampling events, one of the two isokinetic samplers either malfunctioned (4.9 μm; n = 1) or had >5% change in pump flow rate (62.4 μm; n = 2), in which case only one isokinetic sampler measurement was used to calculate sampling efficiency. The coefficient of variation across all isokinetic samplers was 33%.

Table 1 provides the efficiencies for the new sampler and the IPM criterion target for each particle size. Particle sizes 4.9, 9.5, 12.8, 32.7, and 44.3 μm displayed efficiencies that were not significantly different than the IPM criterion [one sample t-test; p(4.9 μm) = 0.68, p(9.5 μm) = 0.79, p(12.8 μm) = 0.054, p(32.7)= 0.33, p(44.3 μm) = 0.17]. Results for the 62.4 and 60.1 μm particles suggest that their means were significantly different from the IPM criterion [one sample t-test; p(62.4 μm) = 0.035, p(60.1 μm) = 0.016]. Figure 4 demonstrates these results as well, indicating that the efficiency of the new sampler matches more closely for particles <50 μm. However, for particles with aerodynamic diameters 62.4 and 60.1 μm, the new sampler performs well below the IPM criterion. Data for CFC and IOM samplers collected using similar methods in a previous study[15] are also shown in Figure 4 for purposes of comparison.

Discussion

The results of this study suggest that the new sampler matches the IPM criterion reasonably well for particles with an aerodynamic diameter of 44.3 μm and below. There is some discrepancy in the efficiency at the 12.8 μm particle size with a low, but technically not a significantly different, efficiency. However, the efficiency for particles that are 62.4 and 60.1 μm in aerodynamic diameter were statistically significantly lower than the IPM criterion. This suggests that the new sampler would substantially under-sample particles larger than ~60 μm, including those up to 100 μm that are still considered inhalable. The transition between matching the inhalable fraction and significantly under-sampling is therefore suggested to occur between 45 and 60 μm.

Compared to the CFC and IOM Samplers (see Figure 4), the newly tested sampler performed well. It showed a better match to the IPM criterion than the CFC, which under-sampled relative to the IPM criterion at all six aerodynamic diameters tested, potentially due to the larger inlet size (15 mm vs. 4 mm). For all sizes <60 μm, the new sampler also better matched the IPM criterion compared to the IOM, which over-sampled relative to the IPM criterion for most particle sizes. This behavior could be explained by the downward orientation of the inlet compared to the IOM's horizontal inlet. The error bars of the IOM measurements also appear larger than those of the new sampler for most sizes as well.

Although the sampler performed well for particles with aerodynamic diameters up to 44.3 μm, it appears that the sampler design did not enable particles with an aerodynamic diameter greater than ca. 60 µm to make that upward turn into the sampler. Fluid dynamics modeling may provide further insight into how these larger particles behave when the sampler is at a 45° downward angle to the horizontal. One issue may be the additional lip on the outer edge of the new sampler face that was added to enable the use of a press-fit transport cap. That lip may be further preventing particles >60 µm from being sampled. However, in workplaces without a substantial exposure potential of these large particles, this sampler may be a viable option for obtaining an estimate of the inhalable particle fraction.

While the new design is somewhat simpler than the IOM, the new sampler is not quite as easy to use as the CFC. Although the inclusion of a single capsule-style filter allows for improved collection of internal wall deposits, the current design is prone to tipping over and potentially losing collected analyte. To keep the sampler from tipping over, extensions could be added that reach down from the side of the new sampler and provide a base to rest securely on a flat surface. Alternatively, a sampler holder could be included with every set of samplers for transport.

Additionally, the transport cap does not fit securely around the inlet, but only fits tightly on the sampler housing. If the sampler were to tip over, the sampled material could relocate from the capsule/filter to the sampler body. Moreover, dislodging the capsule from the top ring can be difficult, especially when using only tweezers. To address potential analyte loss from the capsule, a cap for the capsule-style filter should be developed to aid in ensuring the collected analyte stays in the capsule. The cap material could then be weighed before sampling and included in the gravimetric analysis as well.

Some advantages of the new sampler design are that it is relatively easy to assemble and the press-fit design is less expensive to produce, thereby lowering the cost of the sampler. In fact, this new sampler provides the option of being a disposable sampler due to its low cost.

Conclusion

The new capsule-filter style sampler showed some promise and performed well relative to the IPM criterion for particles up to 44.3 μm in diameter, an extension from the performance of the CFC. However, the new sampler significantly undersampled with respect to the IPM criterion for particles >60 µm and therefore would not be considered a true inhalable aerosol sampler. Despite this performance for particles >60 µm, this sampler performed better than the IOM and CFC sampler for aerosols <44 µm relative to the IPM criterion. Sampler modifications such as elimination of the outer lip or changes in orientation might improve efficiency for the particles >60 µm. A study that focuses on the fluid dynamics of these larger particles and the orientation of a sampler to the horizontal may produce insight for further development of a sampler that matches the IPM criterion at those sizes. The development of a sampler that meets the IPM criterion, is easy to use, is affordable, and that matches the inhalable convention should still be possible.

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