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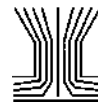
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Investigation of Filter Bypass Leakage and a Test for Aerosol Sampling Cassettes

Paul A. Baron,¹ Aleksandra Khanina,¹ Anthony B. Martinez,¹
and Sergey A. Grinshpun²

¹Centers for Disease Control and Prevention, National Institute for Occupational Safety and Health,
4676 Columbia Parkway, Cincinnati, Ohio

²University of Cincinnati Medical Center, Department of Environmental Health, Cincinnati, Ohio

Plastic filter cassettes (37 and 25 mm), which are press fitted together to seal and hold a filter in place, are commonly used for sampling aerosols. Aerosol bypass leakage around the filter has been reported by several researchers and attempts have been made to test for leakage and to reduce the likelihood of leakage by improving cassette design. Under typical sampling conditions, there is often no indication to the user that leakage may have occurred.

In the present study, a particle count leak test was developed that used a particle counter that measured the particle number concentration of ambient aerosol (primarily submicrometer particles) upstream and downstream of the filter cassette. The relationship between leak test results and particle loss from the filter depended on particle size and type in a complex fashion. The mechanisms of particle loss were investigated and the losses increased for particles above 2 μm and were much greater for solid and fume aerosols than for oil droplets. Although the test could not be used to predict particle mass loss during sampling, the test was a sensitive indicator of cassette bypass leakage and was used to establish compression pressures needed for proper assembly of these cassettes.

INTRODUCTION

Most of the methods for industrial hygiene personal aerosol sampling performed in the United States involve using plastic filter cassettes, which are press-fitted together (National Institute

for Occupational Safety and Health 1994). At least 4 variations of these cassettes are used: 37 mm and 25 mm, either open- or closed-face. All variations use the same filter sealing mechanism. A 37 mm cassette and a 25 mm cassette are shown in Figure 1 that have their cross section colored black. When the cap is not used, the ring seals the edge of the filter and backup pad in place and the cassette is an open-faced sampler. When the ring is replaced by the cap to seal the filter and backup pad, the cassette is a closed-face sampler. Anecdotal evidence indicates that cassettes are commonly sealed by hand pressure (Baron 1998). During normal use, the flow through the cassette is measured before and after sampling to ensure an accurate sampling rate. However, this does not ensure that all the flow passes through the filter, especially if the seal around the edge of the filter is not tightly formed (Van den Heever 1994; Schmidt 1997; Baron 1998). If such bypass leakage occurs, particles that entered the sampler may be lost through the leak and an artificially lowered concentration will be measured, resulting in an underestimate of a worker's exposure. Under most circumstances, there is little or no indication if the cassette was properly assembled or that it leaked.

Attempts have been made using a mechanical press (Frazee and Tironi 1987) or cassette redesign (Van den Heever 1994) to ensure that these cassettes are correctly assembled. In a recent study, the National Institute for Occupational Safety and Health (NIOSH) attempted to produce replicate samples in a chamber using silica dust and 25 mm open-face cassettes (Ashley et al. 1999). Hand assembly of cassettes was performed. While previous work with this chamber indicated that the measured concentration from samples produced in it should have a coefficient of variation (CV) on the order of 0.05 (Carsey 1987), the CVs of sample concentrations measured during the preliminary work of this study ranged from 0.20 to 0.30. This increased variability suggested that leakage occurred in the cassettes because of improper assembly. The range of dust concentrations observed suggested the presence of filter bypass leakage in the cassettes

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Mention of company names or products does not constitute endorsement by the Centers for Disease Control and Prevention.

Address correspondence to Paul A. Baron, NIOSH MS R7, 4676 Columbia Parkway, Cincinnati, OH 45226. E-mail: pab2@cdc.gov

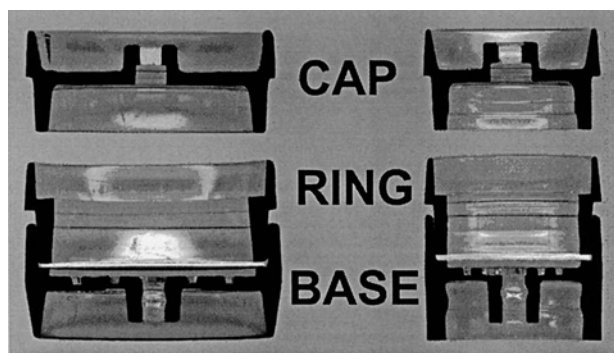


Figure 1. Picture of a 37 mm and a 25 mm cassette cut in half. The cut cassette surfaces are colored black for easier visualization. Bypass leakage can occur around the filter edge where the seal is formed.

of over 50% in some cases. These findings indicated the need for a sensitive test for routine evaluation of cassette assembly.

Several techniques for measuring aerosol penetration through a leak have been reported. Van den Heever (1994) described one for filter cassettes that required a micromanometer. This approach established a baseline pressure drop for each filter type and cassette combination and set limits for an acceptable leak as indicated by a decrease in pressure drop. An unpublished study reported using rubber membranes to replace the filter in a cassette just to evaluate the probability of leakage (Schmidt and Rappaport 1983). A technique to estimate face-mask respirator leakage was developed in which the ambient submicrometer particles were counted upstream and downstream of the respirator (Willeke et al. 1981). The submicrometer particles readily penetrated the respirator along with air passing through a leak, while similarly sized particles carried onto the filter were collected with high efficiency. This approach is now incorporated into a standard respirator fit-testing procedure (Occupational Safety and Health Administration 1998).

The particle count leak measurement technique for respirators was applied to filter cassettes. Although there are similarities in the configuration of the respirator and the sampling cassette, the airflow in the cassette leak is different than in the respirator. The accuracy of the aerosol fraction observed downstream as a representation of the airflow through the leak can be reduced by (a) losses of the particles in the leak and elsewhere in the cassette or (b) by penetration of particles directly through the filter. It is likely that some particles will be lost to cassette walls or in the filter backup pad. Thus the fraction of particles passing through the cassette is smaller than the fraction of air passing through the leak and may change with particle size, sample loading in the cassette, and other factors. However, with the wide range in potential leak rates, even a relatively inaccurate measure of leakage may be useful for quality checks on filter cassette assembly.

Direct submicrometer particle penetration through sampling filters was measured about 20 years ago for several filter types

(Liu et al. 1983). However, filter manufacturers' current specifications indicate that sampling filters having the same nominal pore sizes are much more efficient than those produced at the time of the Liu et al. study. Thus a technique that measures submicrometer aerosol particles downstream of the cassette should measure only particles passing through a leak and thus provide a sensitive indication of leakage in aerosol sampling cassettes.

In a separate study (Baron and Bennett 2001), computational fluid dynamics (CFD) models were developed that described the airflow and particle behavior under a range of leak conditions in a 25 mm open-face cassette. This work indicated that submicrometer particles readily penetrate the leak, while larger particles ($> \sim 2 \mu\text{m}$) impact the filter surface. If larger particles pass through the leak, it is only because they have bounced or become resuspended from the filter surface. Particle bounce is a complex function of particle properties, surface roughness and other properties, the angle of impact, and the velocity of impact (Xu and Willeke 1993; Brach and Dunn 1998). If the sampled particles bounce on the filter surface near a leak, they can be pulled into the leak and not be collected on the filter, thus resulting in an underestimate of mass concentration.

A sampling-cassette leak test was developed using ambient aerosol penetration through cassettes. Such a test would be useful not only to ensure that samplers are properly manufactured and assembled, but also to allow correction of collected mass in leaking cassettes after sampling by laboratories receiving these samples for analysis. In order to determine accuracy of the test and its ability to predict aerosol losses, the test was applied to cassettes using several particle counters, covering a range of leak levels, using several types of challenge aerosol, using several filter materials, and comparing the test results before and after sampling. To understand this leakage/mass loss relationship, which turned out to be complex, the mechanisms of particle behavior within the cassette were also examined.

EXPERIMENTAL

Aerosol Generation and Sample Collection

Experiments were carried out in a laboratory chamber using a fume, a solid particle aerosol, and a liquid particle aerosol to represent some common aerosols that are sampled in the occupational environment. The bypass leakage measurement instruments included 3 different optical particle counter (OPC) models and a condensation particle counter. The leak rate was compared to mass loss for each cassette. In addition, the size-dependent fraction of particles penetrating the leaking cassettes was examined using particle-size spectrometers.

Three types of test aerosol were used: a fume (carbon soot), a dust (Al_2O_3), and a mist (triethanolamine, TEA). Carbon soot was generated using a MAPP gas (methyl acetylene-propadiene, liquified petroleum gas mixture, about $10 \text{ cm}^3/\text{min}$) flame, diluted with 4 L/min of air, passed through an impactor having a cut point of $1.3 \mu\text{m}$, and fed into the collection chamber where it was further diluted. The flame was operated under slightly

oxygen-starved conditions to generate the desired aerosol concentration in the collection chamber.

Aluminum oxide powder (Microgrit LPA 3, Micro Abrasives Corp., Westfield, MA), having a nominal particle diameter of 3 μm (based on Coulter counter measurements according to the manufacturer), was placed in a fluidized bed generator (Model 3400, TSI, Inc., St. Paul, MN).

To produce the oil droplet aerosol, a TEA solution in isopropanol was fed using a syringe pump into an ultrasonic nebulizer (Sono-Tek Corp., Milton, NY). The solution concentration was selected to produce a final TEA droplet distribution after the isopropanol evaporated having a 3 μm particle count mode.

The aerosols were fed into the top of a cylindrical (0.29 m diameter, 1.8 m high) chamber, described in detail by Carsey (1987). The total airflow through the chamber was approximately 160 L/min; the aerosol was mixed with the dilution flow just downstream of a circular mixing disk, which was centered in the chamber and covered 80% of the cross-sectional area. The flow was smoothed by using a honeycomb flow straightener approximately halfway down the chamber. In each run, twenty-seven 25 mm open-face cassettes were loaded with 5.0 μm pore size polyvinylchloride (PVC) membrane filters (Millipore, Bedford, MA) and attached to fittings on a porous plate at the bottom of the chamber.

A real-time aerosol photometer (Model RAM, MIE, Inc., Billerica, MA) was used to monitor aerosol concentration in the chamber. In the case of the aluminum oxide aerosol, the air fed into the generator was humidified to reduce particle charge (Baron and Deye 1990). The interior of the chamber and the exterior of the cassettes were electrically neutralized by exposing them to a ^{210}Po source before sampling. The electric potential of the cassettes was checked with a noncontacting electrostatic voltmeter (Model 3000, Trek, Medina, NY) to ensure neutralization. The chamber walls were checked for charges in a similar fashion. The airflow rate through each sampler was controlled at a nominal 2 L/min, using critical orifices and measured before and after sampling with an automated bubble meter (Gilibrator, Gilian Instrument Inc., Wayne, NJ).

New, unassembled three piece 25 mm cassettes made by the same manufacturer were purchased from several suppliers (Millipore, Bedford, MA; SKC, Eighty Four, PA; Omega Specialty, Chelmsford, MA). Cassettes were assembled using a pneumatic cassette press (Accu-Press, Omega Specialty Instruments, Chelmsford, MA). The closing pressure typically required to produce acceptable assembly, as indicated by the leak test described below, was in the range of 4.1 to $5.5 \times 10^5 \text{ N/m}^2$ (60–80 lb/in²), corresponding to the force range of 330 to 470 N (75–105 lb). When assembling cassettes that were intended to leak, assembly forces of 110–270 N (25–60 lb) were used. It was not possible to reliably produce cassettes with a specific leak rate as indicated by the leak test. Therefore cassettes were assembled using a lower than optimum pressure, and the leak rate was measured until the cassettes having the desired leakage were produced.

The mass of particles collected in each cassette was determined by weighing the filter before and after sampling in a temperature ($70 \pm 2^\circ\text{F}$) and relative humidity ($60 \pm 5\%$) controlled room. Each sample was weighed on a microbalance (Model AT20, Mettler-Toledo, Inc., Hightstown, NJ) capable of 2 μg resolution. Approximately 0.25–0.35 mg of soot, 0.5–1 mg of dust, or 0.25–0.35 mg of oil was collected on each filter. The lower amount of oil and soot was collected to reduce the likelihood of clogging the filter. The tests using aluminum oxide were performed 3 times, and the oil and soot tests were performed twice each.

The size distribution of the soot aerosol had a count median diameter of 0.35 μm (0.68 mass median diameter, calculated using the Hatch-Choate equation) and geometric standard deviation (GSD) of 1.6, as measured by a scanning mobility particle sizer (SMPS; Model 3934C, TSI, Inc., St. Paul, MN). The size distribution of the other 2 aerosols in the chamber was measured using an Aerodynamic Particle Sizer (APS; model 3320, TSI, Inc., St. Paul, MN). The aluminum oxide aerosol mass median aerodynamic diameter was calculated to be 3.6 μm with a GSD of 1.7 from the APS-determined number distribution. Similarly, the TEA aerosol had a 2.9 μm mass median diameter with a GSD of 1.5.

Particle Count Leak Test

The size distribution of the ambient laboratory aerosol was measured using the APS and the SMPS. The mode of the particle number distribution was usually about 100–200 nm (corresponding to the environmental accumulation mode), though concentrations below this size were found to fluctuate significantly from nearby sources, such as pumps or combustion sources. The number concentration of particles dropped off very rapidly as particle size increased, especially above 0.5 μm . Typically, fewer than 10% of the particles were above 1 μm , even when the lower limit of detection was nominally 0.5 μm , as in the case of the APS. These measurements were used to confirm that the majority of laboratory ambient aerosol particles were in the submicrometer range, i.e., likely to pass through a leak and be detectable by the various particle counters used for the particle count leak test. The ambient laboratory aerosol concentration and particle size fluctuated with pollution level and humidity. Higher humidity levels could increase the size of hygroscopic particles, increasing the number of particles that were optically detected.

Three OPCs (Model 217, Model 229A, and Model 229B, Pacific Scientific Instruments, Grants Pass, OR) and a condensation particle counter (PortaCount Plus, TSI, Inc., St. Paul, MN) were used to measure the ambient aerosol penetration of each cassette before and after sampling. Not all of the particle counters were used for each particle count leak test experiment; some were only available for some test runs. Measured particle counter flow rates (with minimum particle size detected in parentheses) were 1.7 L/min for the Model 217 (>0.5 μm); 2.7 L/min for

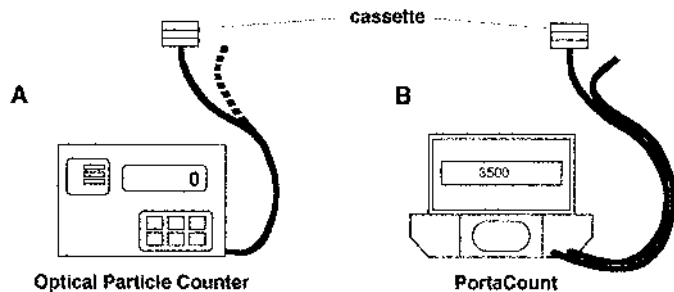


Figure 2. (a) OPC requires 2 measurements—one with the cassette connected and one without the cassette—to measure ambient air. Note that the counter should pull air through the cassette for a minute or two to reach a stable reading, especially after sampling high concentration ambient air. (b) The PortaCount has 2 inlets, one for measuring ambient air and the other connected to the cassette. It can automatically measure both concentrations and display the fit factor.

the Model 229A ($>0.5 \mu\text{m}$); 2.7 L/min for the Model 229B ($>0.3 \mu\text{m}$); and 0.74 L/min for the PortaCount ($>0.02 \mu\text{m}$). The particle counters were generally operated within their manufacturer- indicated operational concentration range, except for the Model 229B. For this model, the 5% coincidence level was expected at an ambient concentration of 70 particles/cm³ (190,000 particles/min). Exceeding this concentration increases the measured bypass leakage level.

Ambient aerosol concentration was determined upstream and downstream of each cassette (Figure 2) before placement in the chamber and after completion of sampling. These measurements were the basis of the particle count leak test. The percent ambient aerosol leakage through the cassettes was monitored using the particle counters and calculated as the ratio of the particle count downstream over the particle count upstream, multiplied by 100.

All 3 OPCs were factory set to perform a count in 1 min, while a total PortaCount measurement lasted 80 s (20 s upstream and 60 s downstream). The ambient OPC measurement was obtained periodically, since this concentration usually remained stable (within about 10%) over 15 min. However, larger variations were noted from day to day, depending primarily on environmental pollution levels. After measurement of ambient air or a high particle concentration, the concentration downstream was measured only after pulling air through the sampler for 2 or 3 min because this was the time needed to clear the OPC detection chamber of previously sampled particles. The PortaCount did not appear to have this problem because it was designed to make rapid comparisons between high and low concentrations. Routine checks of bypass leakage can often be made in a few seconds if no particles are detected downstream of the cassette during this time.

Mass Loss Measurement Procedure

The results of several sample generation runs are reported here. In each run, the filter cassettes were assembled in a press

using a range of pressures to achieve the desired leak rates; the leak test was performed; the cassettes were placed in the chamber and the aerosol sampled; and the sampled mass was determined.

Up to 4 particle counters were used for the leak test during each run. Seven cassettes per run were assembled at optimum pressure (330 N) to ensure no or minimal leakage, as indicated by the particle count leak test. These “nonleaking” cassette samples were used to estimate the true mass concentration in the chamber and to establish the baseline variability for the gravimetric measurements.

Additional Observations and Measurements

The cassettes were examined visually after sampling to provide an indication of the leakage paths. The carbon soot generated using the MAPP gas produced a dark deposit on sample filters and backup pads. Aluminum oxide particle deposits on several filters and cassettes from leaking and nonleaking samplers were inspected using a stereo microscope.

Finally, particle size-dependent penetration through the cassettes was measured using the APS in a smaller chamber with a design similar to that described above. This procedure was the same as that used in other studies to measure particle penetration efficiency of classifiers, such as respirable, thoracic, and PM_{2.5} samplers (Chen et al. 1999; John and Kreisberg 1999; Maynard 1999). Solid, polymethylmethacrylate (PMMA) particles with manufacturer indicated sizes of 3–11 μm were generated using a fluidized bed (John and Kreisberg 1999). The PMMA particles were used to obtain more accurate size distribution data with the APS because these particles had near unit density and were spherical. TEA droplets were generated as described above. Aerosol size distributions from a sampling port within the chamber were measured with and without a 25 mm filter cassette in line; the ratio of these 2 measurements provided the size-dependent fraction of particles penetrating the cassette.

RESULTS

For each of the 27 cassettes in a run, the mass concentration detected for each sample was calculated from the mass measured divided by the product of the flow rate through the sampler and the sampling time. First, the true mass concentration (C_{true}) was estimated as the mean concentration measured using the 7 nonleaking cassettes, i.e., those closed with optimum force. These cassettes were randomly distributed among the 27 cassettes in each sampling run. An analysis of variance (ANOVA) was carried out on the Al₂O₃, soot, and TEA measurements to evaluate the effect of particle type, run-to-run variability, leak test measurement before and after sampling, and the relationship between mass loss and the leak test measurements.

The pooled mass concentration CV for the nonleaking cassettes used for all 3 particle types was 0.048. One outlier in the soot data was dropped because it was greater than 3 standard deviations from the mean of the remaining values. The CV included weighing error and intersampler variability, part of which was the location-to-location variability inside the chamber. The

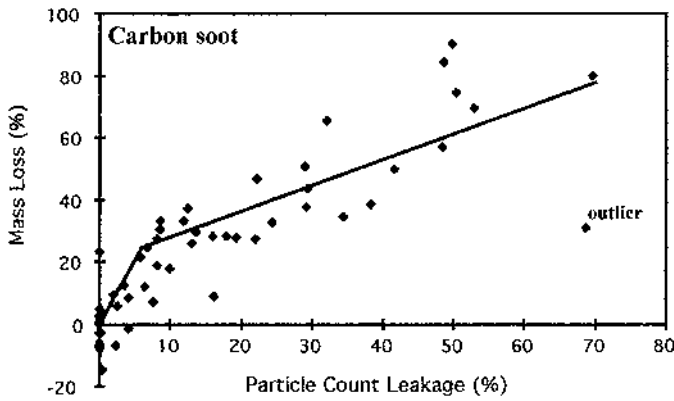


Figure 3. Leakage measured by penetration of ambient aerosol compared to mass of carbon soot dust lost from the filter during sampling. The data presented here are points accumulated from 2 separate measurement runs. Leakage measurement was performed with Model 217 OPC. The fitted line is taken from Figure 4 for comparison.

mass loss in the samples was then determined:

$$Mass\ loss\ (\%) = \frac{C_{true} - C_i}{C_{true}} \quad [1]$$

where C_i is the mass concentration determined from each individual sampler.

A plot of percent mass loss of soot versus percent leakage determined, using the Model 217 OPC, is shown in Figure 3. A similar plot of the Al_2O_3 aerosol data is shown in Figure 4 and of the TEA aerosol data in Figure 5. Because of the clear break in the trend of the Al_2O_3 and soot data, the ANOVA was used to calculate a linear fit in 2 ranges—below and above the break;

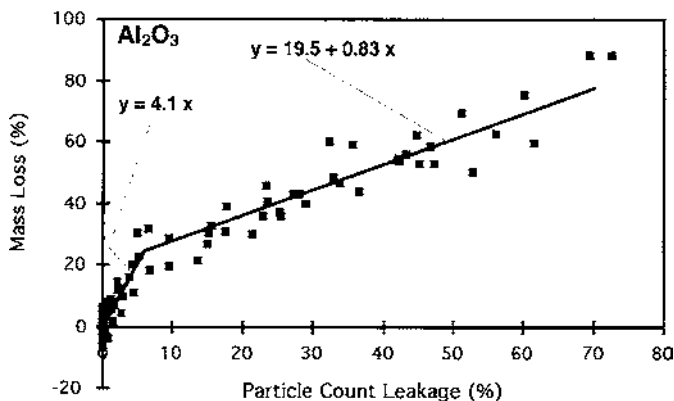


Figure 4. Leakage measured by penetration of ambient aerosol compared to mass of aluminum oxide dust lost from the filter during sampling. The data presented here are points accumulated from 3 separate measurement runs and the regression curve is based on the pooled data. Leakage measurement was performed with Model 217 OPC. The data were fitted in 2 ranges because of the unusual shape of the curve.

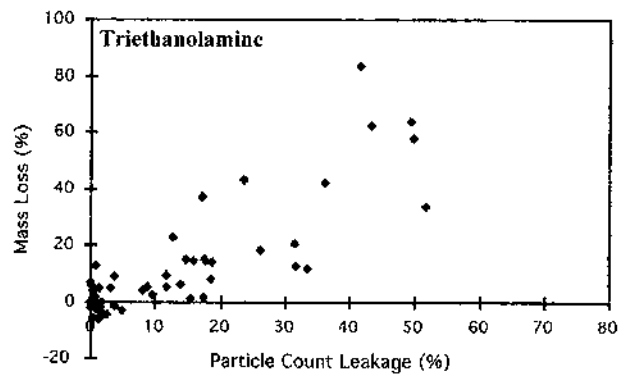


Figure 5. Leakage measured by penetration of ambient aerosol compared to TEA lost from the filter during sampling. The data presented here are points accumulated from 2 separate measurement runs. Leakage measurement was performed with Model 217 OPC.

the break point was selected using the ANOVA for each set of data. The coefficients for the linear fits to the data are indicated in Table 1. The 3 Al_2O_3 data sets and the 2 soot data sets were pooled and the coefficients for the fits to these 2 data sets are also indicated in Table 1. The TEA data were clearly different from the other 2 sets of data; had high variability, especially at high leak rates; and therefore were not fitted using the ANOVA.

For the mass loss versus leak test data, the ANOVA indicated that the run-to-run results changed by a small, but statistically significant, amount and that the effect of the particle counter was not significant. The absolute values of the residual errors for fits to the Al_2O_3 data were examined to see whether they increased with the amount of mass lost. For 2 of the 3 Al_2O_3 runs, there was no significant increase, while for the third run there was a slight increase, having a slope of 0.068 ($p = 0.0032$).

All 4 particle counters were used for 1 Al_2O_3 run and 2 TEA runs and were used to obtain leak test data before and after sampling. For these data, the counter used had a significant effect on the results. It was assumed that there was a linear relationship between the leak test measurements before and after sampling,

Table 1

ANOVA linear equation coefficients (mass lost = $a + b \times \%$ leakage) below and above break point in the curve; the curve at low leak rates is assumed to pass through the origin ($a = 0$)

Run	Break point		a	b
	b	(%)		
Al_2O_3 -1	2.07	18.05	22.04	0.85
Al_2O_3 -2	4.51	4.21	15.12	0.92
Al_2O_3 -3	3.88	7.40	24.08	0.62
Al_2O_3 -pooled	4.10	5.99	19.54	0.83
Soot-1	3.61	5.90	13.46	1.33
Soot-2	2.10	8.18	8.92	1.01
Soot-pooled	3.85	5.94	17.89	0.84

Table 2

ANOVA regression results comparing leak test measurements before and after sampling 2 types of aerosol (% leakage after = $b \times$ % leakage before sampling)

Counter model	Al ₂ O ₃	TEA
MetOne 217	1.32	1.18
MetOne 229a	1.85	0.99
MetOne 229b	0.87	1.01
TSI PortaCount	0.74	1.06

and the results of the regression analysis are in Table 2. The soot data clearly indicated that the measured leak rate after sampling was zero if the initial leak rate was below about 30%. No further analyses of the soot data were performed.

In the smaller chamber, the size-dependent penetration of leaking cassettes was determined using a broad distribution latex or TEA aerosol that was measured upstream and downstream of the cassettes with the APS. Figure 6 shows the size-dependent penetration of oil and solid latex particles through a cassette having a leak rate of 40% (measured with the leak test). The results for several cassettes were similar to those presented in Figure 6. Above 3 μm , the latex particles exhibited significant cassette penetration, indicating particle bounce or reentrainment, while virtually all the larger TEA droplets were collected within the cassette. Note that for both curves, the penetration in the submicrometer range approached that measured with the particle counter leak test using ambient aerosol. The data for other cassettes having smaller leaks had penetrations below 1 μm , similar to the particle count leak test results, and had the impaction cut points at slightly smaller diameters.

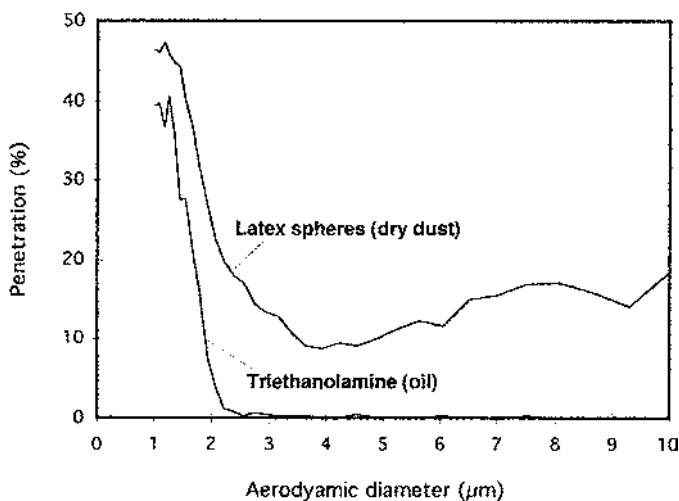


Figure 6. Size-dependent aerosol penetration for oil and solid particles using a cassette that had a leak rate of 40% by the particle count leak test. The larger solid particles penetrated the cassette, indicating particle bounce, while the liquid particles did not, indicating collection somewhere within the cassette.

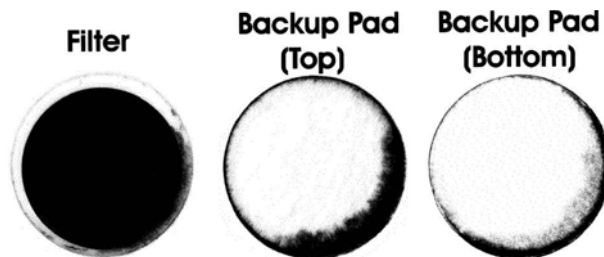


Figure 7. Picture of a filter top surface, backup pad top surface, and backup pad bottom surface after sampling a carbon soot aerosol. The particle count leak test indicated about 50% leakage. The deposits shown here indicate that the aerosol not depositing on the filter (a) passes around the edge of the filter, between the filter and backup pad in the seal region, and down through the backup pad, and (b) beneath the backup pad in the seal region. Most particle deposition occurs when the soot initially enters the backup pad.

The carbon soot aerosol sampled by leaking cassettes could be readily observed at several locations on the filter and backup pad besides on the collection area of the filter. An example of the pattern on a filter and backup pad for a cassette having a high leak rate (50%) is shown in Figure 7. Some soot particles deposited on the top edge of the filter in the seal region. Other deposits were observed on the edge of the backup pad, on top of the backup pad in the seal region between the backup pad and filter, and on the bottom of the backup pad in the seal region. Based on these observations, several likely airflow paths are indicated in Figure 8.

As opposed to the soot aerosol, the Al₂O₃ aerosol was white and visual contrast against the filter was poor. However, using

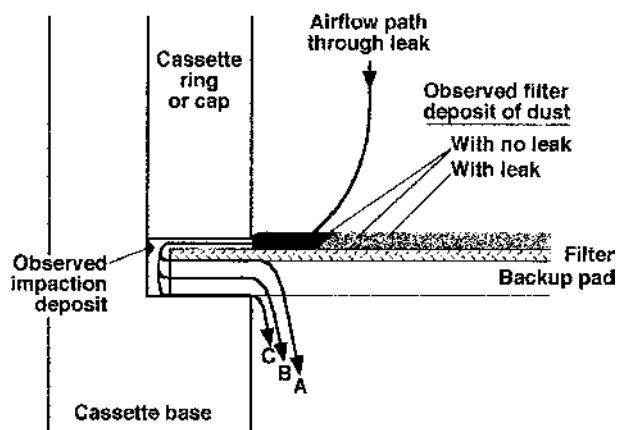


Figure 8. Schematic of filter seal region showing the path of the airflow through the leak. When a leak is present, the observed dust deposit indicates loss of material near the edge of the filter. Some of this lost material is found deposited on the cassette wall. The flow path through or around the backup pad appears to take some combination of Paths A, B, and C, based on soot deposits on the backup pad.

a stereo optical microscope, Al_2O_3 deposits on the filters and cassettes were observed. The filters from cassettes that were well sealed exhibited a well-defined ridge of deposited material near the edge of the filter where the cassette wall contacted the filter surface (see Figure 8). On filters from leaking cassettes, this ridge was generally less well defined, indicating loss of material into the leak. In addition, the leaking cassettes had a distinct powder ring deposited on the outer edge of the cassette base, just above the filter surface (Figure 8). This ring was not entirely uniform around the circumference of the cassette, indicating that the leak did not have a uniform size around the filter circumference.

The leak test was applied to several other types of filters (PVC 5.0 and 0.8 μm , mixed cellulose ester 0.8 μm , polyfluoroethylene 1.0 μm , glass fiber, and quartz fiber) and all appeared to be sufficiently efficient to give zero or very low penetration (<0.1%) through the cassette when the cassette was pressed together with appropriate pressure. Two types of filters (glass and quartz fiber) produced high particle concentrations downstream when small leaks were present. The downstream concentrations decreased over time (as much as a factor of 20 in 10 min). When moderate-sized leaks were present, the downstream particle concentration could be higher than the upstream concentration.

DISCUSSION

Particle Behavior in Leaking Cassettes

The submicrometer particle (ambient aerosol) penetration leak test, which indicated the relative likelihood of leakage in individual cassettes, was used to examine the relationship between the leak rate and particles lost from the filter.

The flow field inside a leaking cassette was described by Baron and Bennett (2001) using CFD calculations and indicated that the airflow makes a bend at the filter surface and enters the leak at high velocity (about 5 m/s) parallel to the filter surface. Thus particles in that flow field must negotiate the change in flow direction to enter the leak. Although dependent on the leak size and mean air velocity in the sampler, particles smaller than about 1.5 μm enter the leak, while larger particles impact on the filter surface. The smaller particles entering the leak can still be collected on the filter surface inside the leak or bypass the filter entirely. Downstream of the filter, a fraction of these smaller particles can be collected on the backup pad or pass entirely through the sampler, in which case they can be detected downstream of the sampler using the leak test.

Effect of Particle Size

For very small leaks, all the submicrometer particles deposit on the edge of the filter. As the leak size increases, up to about 6% by the leak test (pooled results, Table 1), an increasing fraction of submicrometer particles bypass the filter. At larger leak levels, virtually all the particles bypass the filter. This change in aerosol path in the leak results in the large slope at low leak levels of the curves in Figures 3 and 4 and the near unity slope at large leak levels.

The larger particles that impact on the filter surface can bounce off the filter surface, impact other particles and reentrain them, or stick to the filter surface. Because the CFD calculations by Baron and Bennett (2001) indicate that the larger particle trajectories impact the filter surface, the loss of particle mass from leaking cassettes, observed for the 3 types of particles studied, suggests that the particles have been removed by particle bounce or reentrainment. Current theories indicate that particles are more likely to bounce when they are solid, when they approach the surface at high velocities and acute angles, and when the surface is smooth (Xu and Willeke 1993; Xu et al. 1993; Brach and Dunn 1998).

Effect of Particle Type

Of the 3 materials tested, the Al_2O_3 mass loss data in Figure 4 is fitted the best by the regression analysis. The residual variability over the range of leak sizes is almost constant, suggesting that the loss mechanism is relatively simple and well explained in terms of particle bounce. Because the Al_2O_3 particles are sufficiently large and solid, they bounce from the filter surface or are reentrained by impacting particles and enter the leak carried by the high velocity airflow. The loss of particles from the surface of the filter was observed visually, as indicated in the Figure 8 schematic. The observation of particles larger than 2 μm downstream of the cassette (Figure 6) indicates that these particles bounced not only from the filter surface, but also from the cassette wall near the edge of the filter. In both locations, calculated particle Stokes number indicates that these particles should impact the corresponding surfaces.

The soot particles are composed of agglomerated particles of carbon and organic material. The size distribution measured with the SMPS suggests that the mass loss should occur primarily by direct transmission through the leak in a fashion similar to the ambient aerosol. In addition, the impactor placed just after the generator helped to ensure no particles would be large enough to impact on the filter surface. However, agglomerates may have been reentrained from the impaction surface or agglomerates may have formed downstream of the impactor. Sampling these small soot particles, since they were similar in size to ambient particles, was expected to result in a mass loss dependence on particle count leakage that was linear, having a slope of 1 and passing through 0. The shape of the measured dependence (Figure 3) is similar to that for the Al_2O_3 data, although the mass loss at lower leak levels was somewhat lower, suggesting that there was a significant mass of material removed from the filter surface. Because of their loose structure, soot particles are expected to impact the filter surface inelastically and stick. However, their large cross-sectional area will increase reentrainment or shearing of the particles from the filter surface into the leak. The curve in Figure 3 suggests that much of the mass depositing near the leak is lost. However, the variability of the data for larger is somewhat greater than that of the Al_2O_3 data, suggesting that the loss mechanism is more complex and may depend on other factors, such

as the cohesiveness of the particles and the precise shape of the leaks.

The TEA oil droplets appear to impact the filter surface inelastically and stick to the filter at low to moderately high leak levels. Figure 5 indicates that below a measured leak rate of about 10%, all the TEA particle mass is collected on the filter. Above the 10% leak level, droplets do appear to be lost from the filter and penetrate the leak, because significant filter losses are indicated in Figure 5. These losses exhibit high variability and may be a result of particle bounce. For larger leaks, the particles are increasingly accelerated in a radial direction toward the leak entrance. It has been noted that even oil droplets will bounce from a surface when approaching at sufficiently high velocity and at an angle significantly smaller than 90° to the surface (Xu et al. 1993). Although these droplets bypass the filter, they do not appear downstream of the cassette (Figure 6). Impaction on the outer wall of the cassette at the edge of the filter is always at 90° and results in efficient collection at this point.

There is also evidence for several types of particle behavior downstream of the filter. As mentioned above, impaction occurs at the outer cassette wall (Figure 7), as observed visually for Al₂O₃ dust and as indicated by the loss of oil droplets from the filter and their collection somewhere within the cassette. The visual observation of soot particles on the backup pad indicated the particle path downstream of the filter. Although it was expected that particles would penetrate through the edge of the backup pad (Path B, Figure 7) and along the bottom of the backup pad (Path C), a major portion of the particles were observed to deposit on the top of the backup pad (Path A). It appeared that the lack of sealing pressure at the edge of the filter allowed the filter to lift up enough to allow air to pass between the filter and backup pad. There is also indirect computational evidence (Baron and Bennett 2001) that the high velocity airflow through a leak can produce a lift force on the edge of a filter that pulls it away from the backup pad surface. This lift force further enhances the likelihood of Path A as a preferred flow direction. Finally, the proportion of aerosol flow through each leak path may change during sampling as the backup pad becomes loaded with particles. Several leak paths became clogged with soot particles, as was indicated by the efficient collection of ambient particles during application of the leak test after sampling.

Particle losses are expected to occur downstream of the filter, even for submicrometer particles. The backup pad, though having a relatively open structure, can still retain on the order of 30% of the small ambient particles. Thus the particle concentration downstream of the cassette is likely to be in all cases an underestimate of the airflow through the leak.

Particle Count Leak Test

The particle count leak test using ambient aerosol as an indicator of bypass leakage helped to elucidate particle behavior in this study, but it can also provide a quality assurance check on proper cassette construction and assembly. From experience with assembling cassettes in this study, we learned that a fairly

narrow range of pressures (approximately $\pm 20\%$) is needed for leak-free cassettes: too low a pressure produces bypass leakage, while too high a pressure can crack the cassette or cut the filter and produce direct penetration. Since it is difficult to consistently gauge the proper pressure and component alignment when using hand assembly, it is especially important to apply a leak test in this case. A press with a pressure gauge provides more consistent results.

In the Ashley et al. (1999) study, it was found that one batch of cassettes required a much greater than normal force (580 N) for leak-free assembly. Thus use of the test would allow manufacturers to check their cassettes for improper mating during production.

The test assumes that the filter is virtually 100% efficient for all ambient particles passing through the cassette. This appears to be the case for common filter types currently used for sampling. However, binder-free glass and quartz fiber filters may give excessively high downstream readings, even for small leaks. These filters consist of a loosely bound mat of fibers, and the high downstream concentration appears to be caused by fiber shedding from the filter surface in the leak where the air velocity is high.

A second assumption is that the particle losses downstream of the filter do not change greatly when the particle size is below about 1 μm . The leakage was measured with 4 different particle counters and, though slight differences between the counters were noted, these differences were not statistically significant. These insignificant differences support the assumption of size independence, since the counters responded to different portions of the submicrometer size range. The OPCs responded either to $>0.5 \mu\text{m}$ or $>0.3 \mu\text{m}$ particles, depending on the model, while the PortaCount responded to all particles $>0.02 \mu\text{m}$.

Additional leak tests were performed on the cassettes after sampling to determine if the leak changed significantly during sampling. We noted differences when using some of the counters for the dust and oil aerosols. For these types of aerosols, it may be possible to check the cassettes after sampling, though it is clearly desirable to ensure that the cassettes are leak-free before sampling. The data from the soot aerosol makes post-sampling leak testing of the cassettes even more problematic. For leaks smaller than about 30% by the leak test, there was almost no leakage detected after sampling 0.25 mg of soot. Since loss of mass was observed for these cassettes, the soot appeared to build up on the backup pad and prevent further penetration of submicrometer particles through the cassette. Thus if any soot or other agglomerated aerosol is present during sampling, it may prevent ambient particles from penetrating the backup pad, making a post-sampling leak test useless.

Given the variability of mass loss with leak size and particle type and size indicated in Figures 3, 4, and 5, this test would be difficult to use to predict losses in many occupational and environmental situations. However, because the test is sufficiently sensitive and consistent from instrument to instrument, it can be used simply as a benchmark test of proper assembly. While the

particle count leak test has the advantage of being a more direct and sensitive test of leakage than other approaches, such as pressure drop testing (Van den Heever 1994), it has the disadvantage of requiring a somewhat more expensive instrument.

CONCLUSIONS

The particle count leak test presented here provides a direct and sensitive test of cassette bypass leakage and the test results are independent of particle counter type and of filter type (except for loosely bound fibrous filters). The leak test can be used to establish procedures for proper cassette assembly or as a quality control check for cassette production and assembly, resulting in more accurate aerosol sampling. The test cannot be used to predict sample mass lost through a leak because of the loss dependence on sampled particle size and physical state. Investigation of the loss mechanisms indicate the likely fate of each type of particle in a leaking cassette.

REFERENCES

- Ashley, K., Song, R., Esche, C. A., Schlecht, P. C., Baron, P. A., and Wise, T. J. (1999). Ultrasonic Extraction and Portable Anodic Stripping Voltammetry Measurement of Lead in Paint, Dust Wipes, Soil and Air: An Interlaboratory Evaluation, *J. Environ. Monitoring* 1:459–464.
- Baron, P. A. (1998). Personal Aerosol Sampler Design: A Review, *Appl. Occup. Environ. Hyg.* 13(5):313–320.
- Baron, P. A., and Bennett, J. (2001). Calculation of Leakage and Particle Loss in Filter Cassettes, *Aerosol Sci. Technol.* 36(5):632–641.
- Baron, P. A., and Deye, G. J. (1990). Electrostatic Effects in Asbestos Sampling I: Experimental Measurements, *Am. Ind. Hyg. Assoc. J.* 51(2):51–62.
- Brach, M., and Dunn, P. F. (1998). Models of Rebound and Capture for Oblique Microparticle Impacts, *Aerosol Sci. Technol.* 29(5):379–388.
- Carsey, T. P. (1987). LISA: A New Aerosol Generation System for Sampler Evaluation, *Amer. Ind. Hyg. Assoc. J.* 48(8):710–717.
- Chen, C.-C., Lai, C.-Y., Sheng, T.-S., and Hwang, J.-S. (1999). Laboratory Performance Comparison of Respirable Samplers, *Am. Ind. Hyg. Assoc. J.* 60(5):601–611.
- Frazer, P. R., and Tironi, G. (1987). A Filter Cassette Assembly Method for Preventing Bypass Leakage, *Am. Ind. Hyg. Assoc. J.* 48(2):176–180.
- John, W., and Kreisberg, N. (1999). Calibration and Testing of Samplers with Dry, Polydisperse Latex, *Aerosol Sci. Technol.* 31(2–3):221–225.
- Liu, B. Y. H., Pui, D. Y. H., and Rubow, K. L. (1983). Characteristics of Air Sampling Filter Media. In *Aerosols in the Mining and Industrial Work Environments*, edited by V. A. Marple and B. Y. H. Liu. Ann Arbor Science, Ann Arbor, MI, pp. 989–1038.
- Maynard, A. D. (1999). Measurement of Aerosol Penetration through Six Personal Thoracic Samplers Under Calm Air Conditions, *J. Aerosol Sci.* 30(9):1227–1242.
- National Institute for Occupational Safety and Health (1994). *NIOSH Manual of Analytical Methods*, 4th ed. (DHHS/NIOSH Publication No. 94-113), Cincinnati, OH, U.S. Department of Health and Human Services, Public Health Service, Centers for Disease Control and Prevention, NIOSH.
- Occupational Safety and Health Administration (1998). *Respiratory Protection*, Code of Federal Regulations, 29CFR Part 1910.134, January 8.
- Schmidt, A. C. (1997). *Personal Communication*, San Carlos, CA.
- Schmidt, A. C., and Rappaport, S. M. (1983). *An Evaluation of Filter Bypass Leakage in 37 mm Plastic Filter Cassettes*, American Industrial Hygiene Association Conference, Philadelphia, PA.
- Van den Heever, D. J. (1994). Quantification of Bypass Leakage in Two Different Filter Cassettes during Welding Fume Sampling, *Am. Ind. Hyg. Assoc. J.* 55(10):966–969.
- Willeke, K., Ayer, H. E., and Blanchard, J. D. (1981). New Methods for Quantitative Respirator Fit Testing with Aerosols, *Am. Ind. Hyg. Assoc. J.* 42(2):121–125.
- Xu, M., and Willeke, K. (1993). Right Angle Impaction and Rebound of Particles, *J. Aerosol Sci.* 24(1):19–30.
- Xu, M., Willeke, K., Biswas, P., and Pratsinis, S. (1993). Impaction and Rebound of Particles at Acute Incident Angles, *Aerosol Sci. Technol.* 18(2):143–155.