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# Laboratory Measurements of Oil Mist Concentrations Using Filters and an Electrostatic Precipitator

This study investigated the potential for mineral oil mist to evaporate, during sampling, from filters and electrostatic precipitator substrates used to assess personal exposure. If sample evaporation occurs, reported mist concentrations will underestimate true exposure. Mineral oil used as a machining fluid is not normally considered volatile; however, when dispersed as mist its aggregate surface area is so high that significant evaporation can occur. National Institute for Occupational Safety and Health Method 5026 specifies that oil mist concentrations should be determined by collecting mist on filters of mixed cellulose esters (MCE) or polyvinyl chloride (PVC). Collected mist droplets remain dispersed on the filter surface and in contact with passing air while sampling continues, conditions that can lead to sample evaporation. Less evaporation should occur for samples taken with an electrostatic precipitator, where mist droplets are separated from the airflow by electrostatic force and coalesce on the precipitator wall to form a film with relatively low surface area. Collection of mineral oil mist was investigated using a precipitator designed for personal sampling and using either an MCE or a PVC filter. The amounts of oil mist collected using the precipitator were significantly higher than the amounts collected using the filters,  $p < 0.001$ . Further tests in which clean air passed through mist-loaded precipitators and filters showed that the precipitator retained substantially more collected mist than both filters,  $p < 0.001$ , and further suggested that the MCE filter retained more mist than the PVC filter,  $p = 0.059$ . Differences in sample collection and retention between the precipitator and the filters were particularly pronounced at mist loadings below 1 mg.

**Keywords:** evaporation, measurement, mist

**O**il mists from machining operations have long been associated with occupational disease in exposed workers. Concern has increased with recent evidence that exposure to these mists is associated with throat, pancreas, rectum, and prostate cancers,<sup>(1)</sup> as well as with breathing problems and respiratory illnesses.<sup>(2)</sup> As a result the American Conference of Governmental Industrial Hygienists has published<sup>(3)</sup> a notice of intent to reduce the threshold limit value (TLV<sup>TM</sup>) for mists of "mildly refined

oils" from 5 to 0.2 mg/m<sup>3</sup>. Accurate methods to monitor the personal exposure of workers to these mists is important.

Although water-based coolants are increasingly employed and have replaced mineral oil coolants for many applications, oil-based coolants continue to find use. Certain machining operations require straight mineral oils as coolants, whereas other operations use "soluble oil," an emulsion of oil in water. Thus, oil-based aerosols continue to be present in the workplace.

General practice in the industrial hygiene field<sup>(4)</sup> as well as National Institute for Occupational Safety and Health (NIOSH) Method 5026<sup>(5)</sup> employ filters to assess exposure to mineral oil mist. The mineral oils used in some machining operations contain semivolatiles compounds that, during mist collection itself,

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may evaporate from the filter surface.<sup>(6,7)</sup> To the extent that evaporation from a sampling filter occurs, the data obtained may have unnecessary scatter and a bias toward the underestimation of true exposures.

The purpose of this study was to investigate the potential for samples to evaporate from filters and substrates used to collect mineral oil mist. As NIOSH Method 5026 specifies that polyvinyl chloride (PVC) filters or mixed cellulose ester (MCE) filters should be used, they were employed in the present tests. In addition, use of a small electrostatic precipitator (ESP) designed for personal sampling was investigated. In the ESP, mist droplets are first electrically charged and separated from the airstream by electrostatic force, then collected on a cylindrical substrate placed within the collection cylinder. Once collected, the mist in the ESP can coalesce to form a film that is separated from the gas stream and that has much less aggregate surface than the uncoalesced droplets on the filter. Thus, evaporation from the ESP substrate should, in principle, be less than from a filter.

## METHODS

Aerosol was generated by a Collison nebulizer (BGI, Waltham, Mass.), within a 265-L exposure chamber as shown in Figure 1. The concentration of aerosol in the chamber was controlled using a hi-vol sampler (BGI). The hi-vol drew chamber aerosol through a fibrous glass filter to remove excess droplets, then recirculated the cleaned air to the chamber. Airflow through the hi-vol was controlled by adjusting the motor voltage to a constant value that had been established through preliminary experiments.

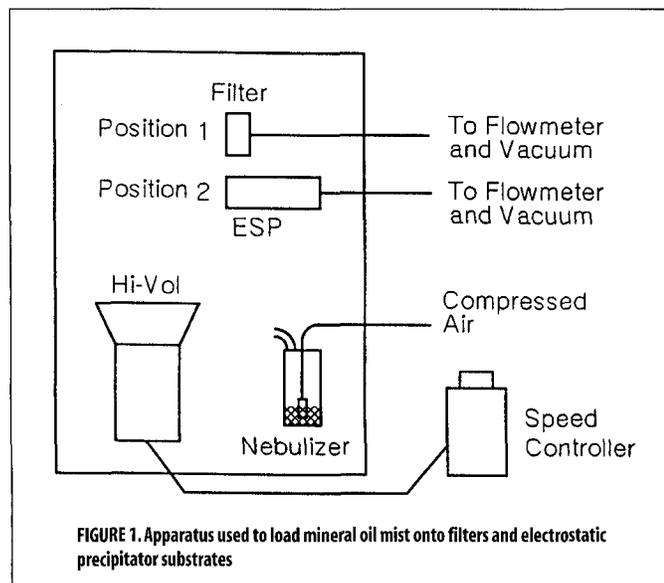


FIGURE 1. Apparatus used to load mineral oil mist onto filters and electrostatic precipitator substrates

The electrostatic precipitator used in this work (Acrosol Associates, Hillsborough, N.C.) was designed for personal sampling at 2 L/min. It had a central ionizing wire within a grounded collection cylinder that was about 7.5 cm long and 1.5 cm in diameter. A disposable collection substrate made from coated aluminum foil was placed within the cylinder for mist collection. The precipitator was powered by a miniature high-voltage power supply that consumed less than 1 watt and could run from a small

rechargeable battery.

To establish that the ESP collected droplets efficiently, it was placed within the exposure chamber while mist was generated. Aerosol at a flow of 2.0 L/min was drawn from the chamber, through the precipitator, and into an Aerosizer (Amherst Process Instruments, Hadley, Mass.), which determined the concentration of droplets in each of 58 size intervals from 0.51 to 7.4  $\mu\text{m}$  in mean size. Similar measurements were then made with the high voltage to the precipitator turned off. A final run was then made with the precipitator turned on once again. The ratio of the average counts with the precipitator voltage turned on divided by the counts with the precipitator turned off determined the fraction of incoming droplets in each size interval that passed through the precipitator. From these data the relationship between droplet diameter and collection efficiency for the precipitator could be determined.

A polystyrene, closed-face filter holder 37 mm in diameter containing a preweighed filter of either PVC or MCE (SKC Inc., Eighty Four, Pa.) and the ESP with a preweighed substrate were placed next to each other in the chamber at Positions 1 and 2, as shown in Figure 1. Flow through each sampler was set at 2.0 L/min and monitored continuously with an in-line rotameter (Dwyer Instruments, Inc., Michigan City, Ind.); each rotameter was calibrated using an automated bubble flow meter (A.P. Buck, Inc., Orlando, Fla.). In each experiment, aerosol was drawn through the filter and through the ESP simultaneously; flow through each was governed using solenoid valves controlled by an electronic timer that had a resolution of 0.1 sec (GraLab Instruments, Centerville, Ohio). The target loadings, designated "low," "medium," and "high," corresponded to loadings for hypothetical samples taken at 2 L/min for about 8 hours at mist concentrations of about 1, 2.5, and 8  $\text{mg}/\text{m}^3$ , to correspond with what might occur in the field. The high-vol speed controller and nebulizer flow were set so that the target amount of mist was drawn through both the filter and the ESP for the same length of time: 60 min for the low and high target loadings and 25 min for the medium loading. After loading, airflow was stopped, the chamber was opened, and the filter and substrate were reweighed to determine the mass of mist collected by each. All weights were measured using a Cahn 27 electrobalance (Cahn/Ventron, Cerrito, Calif.). A balance-room blank was weighed each day to determine the sensitivity of the filter and substrate to changes in balance-room humidity.

To evaluate the potential for samples to evaporate, the loaded filter and substrate were then returned to their samplers and clean lab air was passed through each at 2.0 L/min for 4 hours. The precipitator was turned off during this time. No attempt was made to remove particles from the lab air before it passed through the sampling filters or over the precipitator substrate. As a result the filters would have gained 0.024 mg if lab air contained 50  $\mu\text{g}/\text{m}^3$  of solid particulate; 0.024 mg is a negligible amount compared to the oil mist loadings measured in these tests. The filter and substrate were then removed and weighed once again to determine whether any of the mist previously collected was lost to evaporation during this time. Although workers would not ordinarily pass their day in the exact same exposure pattern as our test program, mist exposure followed by exposure to relatively clean air might occur if workers spent time on the job then moved about the plant, ate lunch, or took breaks.

NIOSH Method 5026 specifies that the amount of oil mist collected on a sampling filter should be determined by infrared analysis. That method is destructive to the sample collected and could not be used in the present tests, where multiple analyses of the collected mist were required.

Paired tests were conducted with the ESP and each of the two filter types for three mist loading conditions and with the filter and ESP in each of the two sampling positions. Each test condition was replicated twice so that in all, 24 experiments were conducted as listed in Table I. Sample blanks for both filters and ESP substrates were used in each experiment, and each sample weight obtained was adjusted for any change in the weight of its corresponding blank. All experiments were conducted in random order. Logarithms of data for loading and evaporation were taken before statistical analysis.

**TABLE I. Experimental Conditions**

Variable	Level Investigated
Sampler type	ESP and MCE, ESP and PVC
Target mist loading	low, medium, high: 1, 2.5, 8 mg
Position in chamber	position 1, position 2
Replicates	2

The mineral oil used in these tests was Metalite SV-5 obtained from a supplier of commercial machining oil (Metal Lubricants, Co., Harvey, Ill.). This oil is comprised of a wide range of compounds, but the most common components are straight-chain, aliphatic hydrocarbons from  $C_{14}$  to  $C_{20}$  in length.<sup>(7)</sup> The size distribution of the oil mist in the exposure chamber was estimated using the Aerosizer while conducting the tests that established precipitator efficiency.

After use, each filter and substrate was placed in its own sealed container. Filters were stored in sealed, plastic dishes, and the cylindrical ESP substrates were stored in plastic tubes with screw caps. To determine the stability of the used filter and substrate weights over several weeks, each used filter and substrate was repeatedly removed from its container, reweighed, then replaced in its container.

## RESULTS

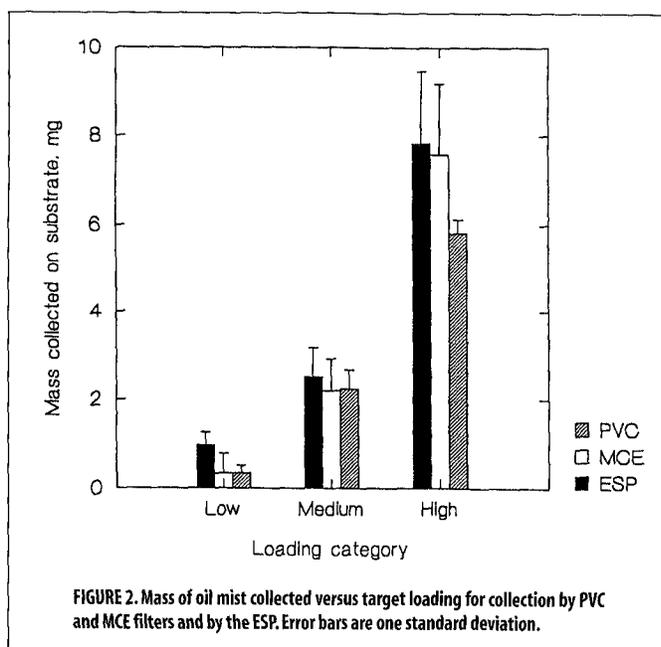
The Aerosizer determined that the mist droplets used in these tests had a count mode of about  $1.5 \mu\text{m}$  and very few droplets larger than about  $8 \mu\text{m}$ . This size distribution was a little smaller than, but not greatly dissimilar to, that measured in industrial machining operations.<sup>(8,9)</sup>

The collection efficiency of the electrostatic precipitator was found to be above 99.5% for droplets from  $0.51 \mu\text{m}$  to  $7.4 \mu\text{m}$ , close to the largest droplets that were generated by the Collison nebulizer. Thus, any substantive differences in collection between the precipitator and the filters was not due to low collection efficiency by the ESP.

Analysis of variance for the data found that the effect of sampler position in the chamber was not significant statistically for amounts loaded, evaporated, or left after evaporation for the ESP substrates or for both types of filters,  $p > 0.21$  for all tests. Thus, the data for

the effect of sampler position were pooled to give a total of four replications for each condition.

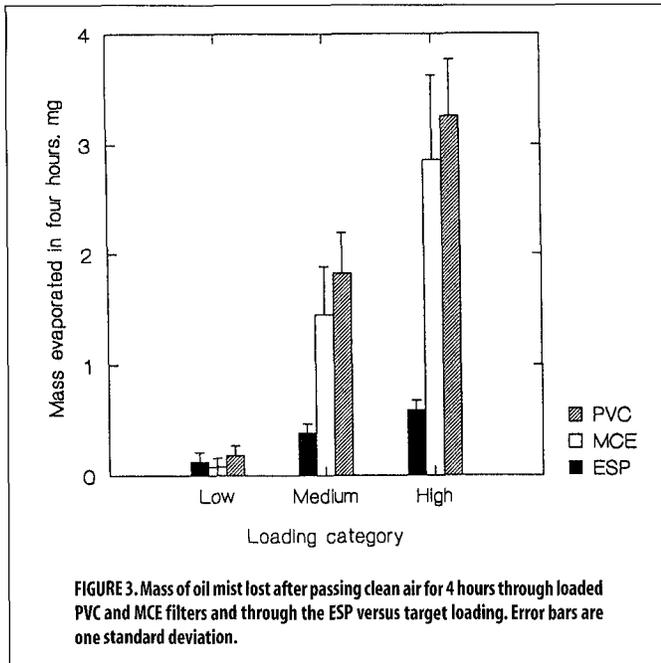
Figure 2 shows, for each loading category, the mass of mineral oil initially collected by the ESP substrate and by both the MCE and the PVC filters. On average for each loading category the ESP substrate collected more mist than the MCE or PVC filters, and a Wilcoxon signed ranks test<sup>(10,11)</sup> found that this difference was significant,  $p < 0.001$ . Although the MCE filter collected more than the PVC filter under the high loading condition, collection for the two filters under other loading conditions was similar. The Wilcoxon test found no significant difference in loading with filter type,  $p = 0.18$ .



**FIGURE 2. Mass of oil mist collected versus target loading for collection by PVC and MCE filters and by the ESP. Error bars are one standard deviation.**

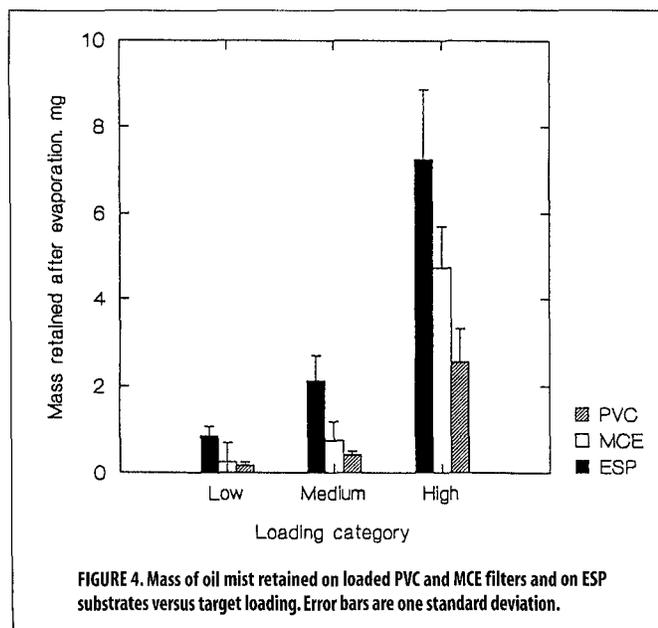
Under the low loading condition the difference between the collection on the ESP substrate and the collection on each of the filters was particularly pronounced. At low loading the ESP collected, on average, 0.98 mg of oil mist, whereas the MCE and PVC filters collected 0.34 and 0.35 mg, respectively, a factor of about 2.8 less.

Figure 3 shows the loss in the mass of mineral oil from the filters and ESP substrates caused by passing clean lab air through each for four hours. These losses, presumably due to evaporation of the collected mineral oil, were substantially greater for the PVC filters than for the MCE filters or the ESP,  $p = 0.033$  and  $p = 0.003$ , respectively. Overall, losses from the filters were significantly greater than losses from the ESP substrates,  $p < 0.001$ , although this situation varied with loading category. For the medium and high loading categories, Figure 3 shows that evaporative losses from both MCE and PVC filters were much greater than the losses from the ESP substrate. For the low loading category this situation changed somewhat, as evaporative losses from the ESP were between those for the PVC filter and the MCE filter. However, for the low category the ESP collected about 2.8 times as much oil as the filters so that more oil was available to evaporate from the ESP substrate. If evaporative losses are expressed as a percentage of initial load, the evaporative losses for both filter types under all loading conditions were substantially



greater than the corresponding losses from the ESP substrates,  $p < 0.001$ .

Figure 4 shows, for each loading category, the mass of mineral oil that remained on the ESP substrate and on both the MCE and PVC filters after clean lab air passed through each for 4 hours. The values in this figure were obtained by subtracting the evaporative losses from Figure 2 from the amounts of oil initially collected from Figure 2. Figure 4 shows that overall the ESP retained nearly twice as much oil as the MCE filters, and over three times as much as the PVC filters; this difference was significant,  $p < 0.001$ . Figure 4 shows that the differences in oil retention among the three samplers were particularly marked for the low loading category, in which the ESP retained 3.3 and 5.0 times as much oil as



the MCE and PVC filters, respectively. Although overall the MCE filters retained nearly twice as much oil as the PVC filters, this difference was of marginal significance,  $p = 0.059$ .

The balance-room blanks were analyzed to determine whether relative humidity affected their weights. Humidity varied from about 25% to over 60% for the period when these experiments were conducted. Regression analysis showed that the weights of the ESP substrates and the PVC filters were stable and had no significant dependence on relative humidity,  $p = 0.91$  and  $p = 0.05$ , respectively. Weights of the MCE substrates significantly increased with increases in humidity,  $p < 0.001$ . This dependence was linear, and had a coefficient of 0.017 mg weight gain per 1% increase in relative humidity. Thus, a 10% increase in balance-room humidity caused an increase in the MCE filter weight of about 0.17 mg, a value comparable to some sample weights in these tests and similar to weights expected from 8-hour samples close to the proposed TLV of 0.2 mg/m<sup>3</sup>. If a nongravimetric method of analysis such as the infrared procedure specified in NIOSH Method 5026 is used to determine oil mass, then humidity will have no effect on filter and substrate weights. In practice, however, gravimetric analyses are also used.<sup>(5)</sup> In these cases susceptibility to humidity effects is important.

Reweighting used substrates and filters over several weeks showed that all gradually lost mass over time. For each day after sampling spent in a sealed container, the ESP substrates lost about 0.8% of the mass originally collected, and this dependence was significant,  $p = 0.01$ . Both filters lost approximately 0.5% of the mass they originally collected for each day after sampling, and this dependence was also significant,  $p = 0.01$ . The loss coefficients of 0.8%/day for the ESP substrates and 0.5%/day for the filters were not significantly different. For both the ESP substrates and for the filters, the percentage loss depended significantly on the initial mass load,  $p < 0.02$  for both, with the highest percentage losses in cases where the initial loading was low, and the lowest percentage losses when the initial loading was high. Thus, delay in analyzing a sample after it is taken may reduce the resultant concentration reported, with the greatest percentage reductions associated with the lightest substrate loadings.

## DISCUSSION

Data given in Figures 2 and 3 support the idea that sample evaporation can occur, particularly when samples of mineral oil mist are taken using MCE and PVC filters. Figures 2 and 4 suggest that differences in sampling methods for oil mist can be especially important when measured mist concentrations are low, less than about 1 mg/m<sup>3</sup>. Evaporation from ESP substrates was substantially less than evaporation from either filter type, presumably because the ESP removes collected mist from the airstream and coalesces it to form a film with less surface area.

Figure 2 shows that significantly less oil was collected by the filters than by the ESP substrates. These results were obtained in tests where the filters and substrates were loaded quickly, in 1 hour or less. In the field, sampling would occur over several hours so that the opportunity for sample evaporation would be greater. Thus, the differences between sample loadings for the filters and ESP substrates as shown from the lab results in Figure 2 might be more pronounced for field conditions where the filters and

substrates are loaded more gradually.

Figure 3 shows that evaporative losses from loaded MCE and PVC filters during exposure to clean air were more substantial than losses from a loaded ESP substrate. To the extent that a worker enters clean areas while wearing a personal monitor, similar evaporative losses could occur in practice.

Figure 4 shows residual retention after collection and evaporative losses. A comparison of the filter types shows that retention by the MCE filter was better than retention by the PVC filter; however, the weight of the MCE filter was sensitive to changes in lab humidity. Thus, both filter types had shortcomings in these tests. The ESP substrates were better at sample collection and lost less in evaporation to clean air; thus, they had greater retention than either filter. In addition, the ESP substrates had stable weights despite humidity fluctuations in the balance room.

The results presented here suggest that mist samples are gradually lost over time when stored. Perhaps the collected mist re-partitions from the filter or substrate to the inside surface of its storage container, to the air within the container, or both. These results suggest that excessive delays in sample analysis may lead to the underestimation of sample concentration. The test methodology used here may have aggravated this trend, as repeated opening of the sealed containers to reweigh the filters or substrates would cause repeated opportunities for replacement of vapor-saturated air in the container with unsaturated lab air.

The mineral oil used in this work was fresh. In a manufacturing plant the oil is recirculated, with periodic makeup to replace carry-over due to parts transfer, so that the average age of the used mineral oil would be greater than that tested here. If the lighter fractions of this oil evaporate during use,<sup>(7)</sup> then mist generated from this oil would be deficient in the more volatile fractions and less prone to evaporate. By this reasoning, the lab results reported here would overestimate the tendency for sample evaporation in the field. In addition, the chemical composition of mineral oil will vary according to its proposed use and from manufacturer to manufacturer.

Evaporation rate also depends on the oil vapor concentration of the surrounding air calculated on a compound-by-compound basis; these concentrations will increase with temperature but decrease with dilution by outside air. Because the lab tests reported here were conducted within a closed chamber, the oil vapor concentrations should have been relatively high and decreased the tendency for evaporation. During summer, warm temperatures in a plant should increase vapor concentrations; however, dilution by outside air to control temperature will reduce vapor concentrations; the reverse will occur in winter. The degree to which any of these factors plays a role at a particular plant will depend on plant ventilation practice, temperature, oil properties, and the nature of the machining operations for which the fluid is used. Thus, the present results indicate the possibility that sample evaporation can occur, but the quantitative results presented here cannot reliably be used to predict the degree of evaporation that might occur in the field. Results in this paper are presented as sample masses rather than as concentrations to discourage extrapolation of the quantitative results to concentration measurements made in the field.

Evaporation of collected oil mist could lead to scatter in exposure assessment data taken in the field using MCE or PVC filters. Data scatter would be aggravated if evaporation varies from location to location due to factors such as variable ventilation rates,

temperature conditions, or fluid makeup. Such scatter would tend to obscure any relationship between exposure and disease. Whether this problem has occurred in past studies is difficult to say, but future studies should consider alternative sampling techniques such as ESP to minimize the problem.

## CONCLUSIONS

The vapor pressure of mineral oils used as coolants in metal machining operations is so low that evaporation of pooled liquids does not occur quickly; however, these materials can experience significant evaporation when they are dispersed as an aerosol of small droplets with high specific surface. Overall, for the sampling protocol used here, this work found that a small electrostatic precipitator retained nearly twice as much oil mist as an MCE filter and over three times as much as a PVC filter. Retention was greater in the precipitator because it separates collected mist from the airstream and coalesces it into a film that has much less aggregate surface area than that for the aerosol droplets collected on a filter. The quantitative results presented are particular to the lab tests used and cannot reliably be extrapolated to field conditions where sample retention could be greater or less. Nevertheless, these results do show that evaporation from a sampling filter can occur, and suggest that evaporation can be minimized by collecting samples using an electrostatic precipitator.

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