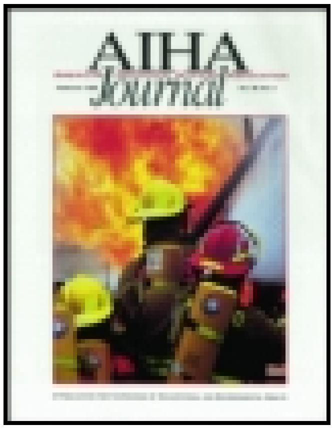

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"Worst-Case" Aerosol Testing Parameters: III. Initial Penetration of Charged and Neutralized Lead Fume and Silica Dust Aerosols through Clean, Unloaded Respirator Filters

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"Worst-Case" Aerosol Testing Parameters: III. Initial Penetration of Charged and Neutralized Lead Fume and Silica Dust Aerosols through Clean, Unloaded Respirator Filters

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The National Institute for Occupational Safety and Health (NIOSH) tests and certifies respirator filter media according to Title 30, *Code of Federal Regulations,* Part 11 (30 CFR 11). Subpart K of those regulations specifies that a silica dust test, silica mist test, and/or lead fume test will be used to test and certify dust and mist; and dust, fume, and mist particulate air-purifying respirator filter media. NIOSH studies have shown that an aerosol particle of a certain size can be identified as the most penetrating particle ("worst case") size. Commercial filter media of various types have been studied and the filter's performance against a worst-case sodium chloride (NaCl) and dioctyl phthalate (DOP) aerosol evaluated. This investigation was done to complement those previous studies by determining how one manufacturer's particulate filters performed against the existing certification aerosol challenges as compared with the worst-case size DOP and NaCl aerosols. Only initial penetration values were determined, and no loading effects were considered. Both neutralized (Boltzman charge distribution) and unneutralized aerosols were used in order to assess the contribution of charging. The results show the dramatic effect of particle size on filter efficiency, and they show that the present methods are not as sensitive as the worst-case aerosol method.

Introduction

The National Institute for Occupational Safety and Health (NIOSH) currently tests and certifies respirator filter media in accordance with the regulations set forth in Title 30, *Code of Federal Regulations,* part 11 (30 CFR 11).[1] Subpart K of those regulations specifies that a silica dust, silica mist, lead fume, and/or dioctyl phthalate (DOP) aerosol be used to test respirator filter media. Presently only high efficiency (HE) filters are certified with an approximately 0.3-μm DOP aerosol, but a 0.3-μm DOP aerosol's use for other filter classes is under consideration. Alternative challenge aerosols being considered by NIOSH include a solid aerosol and a liquid aerosol. These changes have been put forth to bring respirator filter testing up to date with current technologies. Some of these changes, based on "worst-case" testing parameters have been reported earlier.[2]

This study was undertaken to determine the initial instantaneous count efficiencies of dust and mist (DM); paint, lacquer, and enamel mist (PLEM); and dust, fume, and mist (DFM) respirator filter media against the certification silica dust and lead fume aerosols. The filter efficiency test system (FETS)[3] was used. These data are compared with earlier count efficiency results for these respirator filters against the most penetrating worst-case size DOP[2] and sodium chloride (NaCl)[2] aerosols. Thus, a direct comparison of the initial count efficiency values employing the certification aerosols and the worst-case aerosol was possible. Some additional experiments with "neutralized" (Kr-85 radiation source) silica dust and lead fume aerosols were performed in order to assess filter collection effects caused by aerosol charge.

Background

An earlier study by Reed et al.[4] compared the filter penetration of NaCl, DOP, silica dust, and lead fume on a variety of filter media. Also, some initial results on relative humidity and charging effects were presented. That study suggested that DOP and NaCl would be reliable aerosols for measuring the efficiency of respirator filter media and their degradability. The results of that study showed the following conclusions.

(1) The silica dust test is not a sensitive indicator of the relative efficiency of respirator particulate filters since the penetration of all filter types ranged from 0.06% for HE filters to 0.07% for DM filters. This was attributed to the large size of the silica dust particles as specified in the regulations.

(2) The lead fume test seemed to be somewhat more differentiating than the silica dust test. In this case the DM filters showed 8.8%-14.2% penetration while the DFM and HE filters gave penetrations of $< 0.63\%$. The differences in the DFM and HE filter results were not statistically significant.

(3) The lead fume and silica dust aerosols were difficult to generate and maintain at a stable mass concentration and particle size over the entire length of the test runs (90 min for silica dust and 312 min for lead fume).

(4) The instantaneous penetration of the silica dust and lead fume aerosols could not be determined at any given time during the test since these efficiencies are a time-weighted average (TWA) over the total test time as determined gravimetrically.

The above study suggested that smaller, more penetrating aerosols are more vigorous, discriminating challenges for respirator filter media testing.

Experimental Materials and Methods

The silica dust and lead fume aerosols used are exactly as described in 30 CFR 11 Subpart K (11.140-4, "Silica Dust Test" and 11.140-6, "Lead Fume Test") and were not subjected to any size classification or separation. The 30 CFR 11-140-4 silica dust particle-size criteria are as follows: geometric mean of 0.4–0.6 μm and a standard geometric deviation (σg) not to exceed 2. The lead fume aerosol is generated by impinging an oxygen gas flame on molten lead (30 CFR 11-140-6 [d]) with no particle size or σg being specified. To facilitate a direct comparison to the worst-case size aerosols, both the silica dust and lead fume aerosols were sized by independent methods.

The aerosols generated in the certification test chambers were withdrawn, diluted to $\simeq 2 \times 10^4$ particles/cc, and employed for testing. The dilution of these aerosols with diluter air filtered through high efficiency filters should not alter the size characteristics of the aerosol. For the aerosol "neutralization" studies, the only experimental difference was that a Kr-85 radioactive source was placed in line to reduce the aerosol's charge to a Boltzman distribution.

DM, PLEM, and DFM filters from a single lot of Manufacturer A's filters[2] were tested. Individual filters were tested "as received" at flow rates of 16 ± 0.3 L/min and 85 ± 1.4 L/min. The initial instantaneous penetration was determined 2 min after exposure to the challenge aerosol by means of the TSI, Inc., Filter Efficiency Test System (FETS) (TSI, Inc., St. Paul, Minn.) which has been described by Remiarz et al.[3] In the present study only one respirator was tested at a time since the initial instantaneous aerosol penetration was the parameter of primary interest. A simplified schematic diagram of the FETS as used in this study is shown in Figure 1.

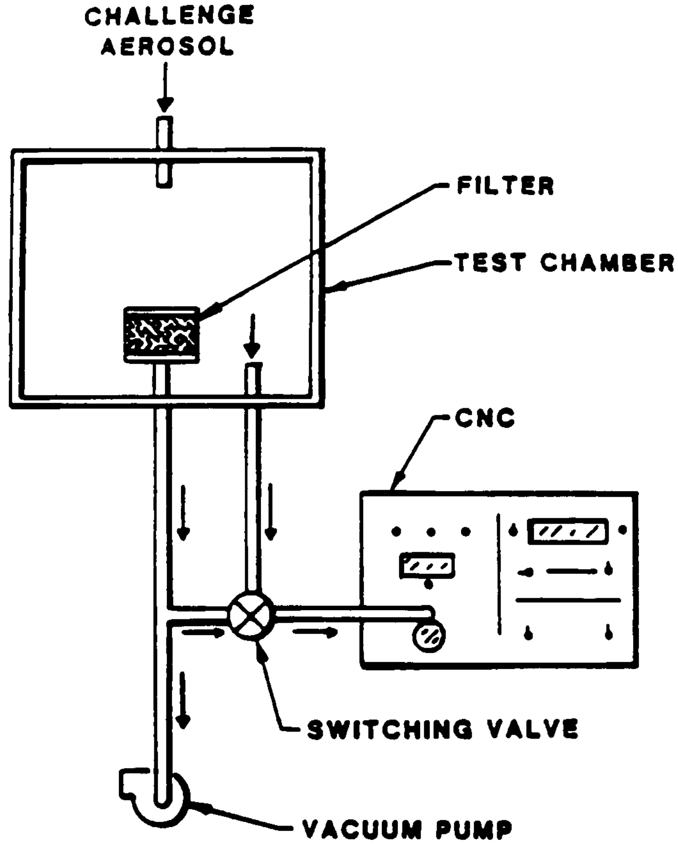

Figure 1—Operating schematic of TSI Filter Efficiency Test System.

Results and Discussion

The initial instantaneous percent count efficiency values for the DM, PLEM, and DFM filters studied against the certification aerosols are presented in Table I. The silica dust data show that the DM filters at 16 L/min (equivalent to 30 CFR Part 11 requirement for this filter configuration) gave an initial instantaneous count efficiency of 98.12%. Presently DM filters are required to have a mass efficiency of > 99% when tested according to the silica dust test (30 CFR 11.140), which is a 90-min, time-averaged gravimetric test. When the test flow was increased to 85 L/min, the efficiency against the silica dust decreased to 96.03%. A similar trend was seen with the PLEM and DFM filters against the silica dust aerosol (Table I). In each case a significant decrease in efficiency was seen with increased test flow rate. It appears, however, that the magnitude of this change is less dramatic for the DFM filters than for the DM or PLEM filters. This same effect was seen with the lead fume challenge aerosol

TABLE I
Initial Instantaneous Filter Efficiency for "Worst-Case" Size Aerosols and Silica Dust and Lead Fume Aerosols

		Silica Dust Data[A]		Lead Fume Data[B]		Worst-Case Data	
Filter Type	Flow Rate (L/min)	# Filters Tested	% Efficiency (Standard Deviation)	# Filters Tested	% Efficiency (Standard Deviation)	% Minimum Efficiency	Particle Size Region (μm)[C]
DM	16	22	98.12 (0.67)	10	91.64 (1.33)	87–89	0.04–0.08
	85	6	96.03 (0.58)	7	80.11 (0.86)	69–75	0.04–0.10
PLEM	16	20	99.36 (0.07)	10	91.82 (0.82)	88–93	0.07–0.12
	85	6	96.11 (0.25)	5	81.63 (1.29)	76–78	0.06–0.10
DFM	16	10	99.42 (0.16)	13	98.23 (0.29)	98–99	0.06–0.10
	85	6	98.49 (0.18)	7	89.13 (0.98)	87–92	0.05–0.10

[A]Silica dust size of 0.48 μm, determined by SEM.
[B]Lead fume size of 0.15 μm, determined by DMPS.
[C]Count mean diameter, determined by DMPS.

and was more pronounced. Against lead fume, the DM filters gave an initial efficiency of 91.64% at 16 L/min. When the test flow rate was increased to 85 L/min, the efficiency decreased to 80.11%. This significant flow rate effect likewise was seen with the PLEM and DFM.

Upon comparing these data, it can be seen that in all cases the lead fume aerosol gives lower filter efficiency values than the corresponding silica dust results. For example, the DM filters gave efficiencies of 98.12% at 16 L/min and 96.03% at 85 L/min against silica dust as compared to 91.64% at 16 L/min and 80.11% at 85 L/min against lead fume. Table I presents similar data for PLEM and DFM filters. These data are consistent with the earlier study by Reed et al.[4] which show that the lead fume test aerosol is a more sensitive and differentiating indicator of the relative efficiency of particulate filters than the silica dust aerosol. It must be remembered, however, that presently DM filters are not required to be tested and pass a lead fume challenge.

The present certification program requires DFM filters to have a mass efficiency of > 99% when tested according to the lead fume test, which is a 312-min, TWA gravimetric test (30 CFR 11.140-6), with no instantaneous efficiency values being obtained. Thus, the DFM filters are designed to protect against fume aerosols which are smaller than silica dust. This is why the DFM filter results gave the highest efficiencies of those tested and did not show the significant magnitude of differences in efficiency when going from silica dust to the lead aerosol challenge. The data show that the DFM filters at 16 L/min (equivalent to 30 CFR 11 requirements for this filter configuration) gave an initial instantaneous count efficiency of 98.23%. When the flow rate was increased to 85 L/min, an initial instantaneous efficiency value of 89.13% was obtained with the lead fume aerosol. As anticipated, when the smaller particle-size lead fume aerosol (0.15 μm count mean diameter [CMD], σg of 1.92, determined by differential mobility particle sizer [DMPS] assuming a log-normal distribution) was used for testing, rather than the silica dust aerosol, the DM and PLEM percent efficiencies dropped considerably: 16 L/min DM filter efficiency dropped from 98.12% to 91.64%, and the PLEM filter efficiency dropped from 99.36% to 91.82%. This is dramatized further when one looks at the 85 L/min data where the DM efficiency went from 96.03% to 80.11% and the PLEM efficiency dropped from 96.11% to 81.63%.

The silica dust results also can be compared with the worst-case results presented for DOP and NaCl in Part I of this series[2] (Table I). It can be seen that at 16 L/min, the initial instantaneous count efficiency results with silica dust are about 9%-11% higher than those obtained using a worst-case size aerosol.[2] The results obtained with the worst-case challenge aerosol at 85 L/min are from 21% to 27% lower than with silica dust. These results indicate (1) the worst-case aerosol is a more critical, vigorous test aerosol and (2) that the percent efficiency for worst-case aerosols decreases more rapidly with increasing flow rate than for the larger silica dust aerosol. The PLEM and DFM filters gave higher efficiency values than the DM filters. The PLEM filter gave efficiencies that were 6%-11% and 18%-20% lower than the worst-case aerosols for 16 and 85 L/min, respectively. The DFM filters, which should be the most efficient against smaller fume particles, did possess the best filter efficiency characteristics of those studied, with efficiency differences that were as low as 0.42% for 16 L/min and 6.5% for 85 L/min. These efficiency differences are caused by (1) the silica dust's particle size (0.48 μm by scanning electron microscopy [SEM]), which is significantly larger than the worst-case aerosol particle size, and (2) aerosol charge differences, remembering that the worst-case aerosols are neutralized.

Likewise, the lead fume results can be compared with the worst-case DOP and NaCl results. The lead fume data show that the DFM filters at 16 L/min gave an initial instantaneous count efficiency of 98.23%, whereas the worst-case particle-size aerosols[2] gave 98%-99% efficiency at 16 L/min. When the flow rate was increased to 85 L/min, an initial instantaneous efficiency value of 89.13% was obtained with the lead fume aerosol and compare with worst-case efficiency values of 87%-88% for DOP and 91%-92% for NaCl.

Further, comparison of the worst-case initial instantaneous count efficiency with the results for the silica dust and lead fume aerosols reveal some interesting data trends. First, there are greater differences in the initial efficiency results when the silica dust results are compared to the worst-case aerosol results than when the lead fume results are compared to the worst-case results. This is probably because of the aerosols' size differences, with the worst-case aerosol being smaller than the lead fume aerosol (0.15-μm CMD and σg of 1.92, determined by DMPS—the top region of the worst-case aerosol size), which in turn is smaller than the silica dust aerosol (0.48 μm, determined by SEM). This is exemplified by the fact that the differences in the efficiency magnitudes from the worst-case challenge aerosol are DM > PLEM > DFM, which correlate with the challenge aerosol's particle sizes: silica dust larger than lead fume larger than worst case. This is demonstrated further by the fact that a direct comparison of the two certification test aerosols versus the worst-case size aerosol revealed that for the filters tested, there is a significant difference for the silica dust, and there is not sufficient evidence to detect a difference for the lead fume aerosol (paired t-test on ln transformed data at $\alpha = 0.05$). Secondly, there is a significant decrease in percent efficiency for both the silica dust and lead fume aerosols as the flow rate is increased from 16 to 85 L/min. Thus, both particle size and flow rate have a significant effect on the initial instantaneous filter efficiency values determined for commercially available respirator filters.

The second part of this study examined the effect of aerosol charge on the filter's initial penetration by silica dust and lead fume aerosols. A Kr-85 radiation source was placed in line to reduce the silica dust and lead fume aerosol charge to a Boltzman distribution. The results obtained for the charge conditioned/neutralized certification silica dust and lead fume aerosols are presented in Table II, along with the results of the "charged" aerosols from Table I. Table II also indicates the statistical significance of the differences. It can be seen that the effect of charge on these efficiency values is less than or equal to 5% for both aerosols. Also, the aerosol charge effect appears larger for the silica dust than for the lead fume and is reflected by the relative differences in the efficiency values, where the differences for the silica dust

TABLE II
Initial Instantaneous Filter Efficiency Data for "Charged" and "Neutralized" Silica Dust and Lead Fume Aerosols

		Silica Dust Data			Lead Fume Data		
		Charged[A]	Neutralized		Charged[A]	Neutralized	
Filter Type	Flow Rate (L/min)	% Efficiency (Standard Deviation)	# Filters Tested	% Efficiency (Standard Deviation)	% Efficiency (Standard Deviation)	# Filters Tested	% Efficiency (Standard Deviation)
DM	16	98.12 (0.67)	21	98.67 (0.40)[B]	91.64 (1.33)	10	93.12 (0.90)[B]
	85	96.03 (0.58)	7	94.35 (1.40)[B]	80.11 (0.86)	5	80.93 (1.34)
PLEM	16	99.36 (0.07)	24	98.83 (0.21)[B]	91.82 (0.82)	10	92.98 (0.75)[B]
	85	96.11 (0.25)	5	91.04 (0.65)[B]	81.63 (1.29)	5	79.76 (1.22)[B]
DFM	16	99.42 (0.16)	10	99.33 (0.16)	98.23 (0.29)	13	97.78 (0.50)[B]
	85	98.49 (0.18)	5	95.53 (0.15)[B]	89.13 (0.98)	7	88.50 (1.01)

[A]Charged data from Table I.

[B]Significant difference between charged and neutralized data.

range from -0.55% to +5% and the lead fume differences range from -1.48% to 1.87%. This probably is caused by the aerosol generation methods and/or to the aerosols' ability to retain a higher charge. The evaporation/condensation aerosol generation procedure of the lead fume aerosol would be expected to provide a smaller charge than the fluidized bed-type, generated silica dust aerosol.

To further investigate the charge effects, some preliminary tests were obtained using a neutralized and nonneutralized DOP challenge aerosol. Two separate runs were done at each particle size for each filter: one using a "charged" or nonneutralized DOP aerosol and the other using a neutralized DOP aerosol. These preliminary results revealed that aerosol charge had a large effect on the efficiency of filters when tested against a worst-case DOP aerosol at 85 L/min. The efficiencies determined for the DM filter were 85.90% efficiency for a nonneutralized DOP challenge versus 75.03% efficiency for the neutralized DOP challenge (0.045 μm) produced as per Stevens and Moyer.[2] The difference in efficiencies was 10.87%. The difference in efficiencies determined for a slightly larger DOP aerosol (0.061 μm) was not as significant (81.39% efficiency for nonneutralized versus 76.73% efficiency for the neutralized, or a difference of 4.66%). Similar results were obtained for a PLEM filter run against the 0.045- and 0.061-μm DOP aerosols which were neutralized and nonneutralized. Those results are 87.14% efficiency for the nonneutralized versus 77.25% efficiency for the neutralized, representing a difference of 9.89% with the 0.045-μm DOP challenge. The PLEM results with the 0.061-μm DOP are as follows: 82.5% efficiency, nonneutralized, versus 78.49% efficiency, neutralized, or a difference of 4.01%. Thus, a 4%–11% increase in percent efficiency resulted when the aerosol was not neutralized with the Kr-85 source to reduce the aerosol's charge. Also, it appears that the effect of the aerosol's charge on percent penetration is related to and dependent on the aerosol's particle size.

Conclusions

This study shows that particle size has a dramatic effect on the experimentally determined initial count efficiency of respirator filter media. As the particle size is reduced in going from the silica dust to the lead fume aerosol, the filters' percent efficiencies decrease. This indicates that the lead fume is more penetrating and, thus, more of a discriminating test aerosol than silica dust. Also the worst-case particle-size aerosol gave significantly lower efficiency values for all filter types and flow rates, except for the DFM filters tested at 16 L/min. This confirms that the worst-case aerosol is more penetrating and a more discriminating test aerosol than either silica dust or lead fume. Additionally, the effect of aerosol charge on the percent filter efficiency was investigated. The effect of charge reduction on the silica dust and lead fume aerosol was not nearly as large as the charge effect observed with the worst-case particle-size aerosol. Finally, the differences seen in the percent penetration between the 16 L/min and 85 L/min data was substantial. These flow results are in agreement with earlier NIOSH[2] findings.

The findings from Reed et al.,[4] when considered in conjunction with the results from this study, indicate that smaller, more penetrating aerosols would be more discriminating and would make a better test system. It should be noted, however, that these studies all relate to attempts to find respirator filter tests that are or relate to worst-case testing criteria and are not necessarily indicative of measured efficiencies against a "workplace-type" aerosol.

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