

COMPARISON OF RESPIRATOR PARTICULATE
FILTER TEST METHODS

by

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ABSTRACT

Four brands of respirator particulate filters were tested for penetration using di-octyl phthalate (DOP), silica dust (SiO_2), lead fume (PbO), and sodium chloride (NaCl) aerosols. Results suggest that DOP and NaCl would be reliable aerosols for measuring the efficiency and degradability of respirator particulate filters. A recommendation has been made to control the relative humidity and charge of the NaCl aerosol.

INTRODUCTION

Currently, NIOSH tests and certifies respirator filter media according to the provisions of Title 30, Code of Federal Regulations, Part 11 (30 CFR 11). Subpart K of these regulations specifies the use of silica dust, silica mist (SiO_2), lead fume (PbO), and $0.3\ \mu\text{m}$ di-octyl phthalate (DOP) as challenge agents for testing particulate air-purifying respirator filter media. Challenge aerosols which have been and are being considered as alternatives to the above tests include a solid aerosol, (e.g., sodium chloride (NaCl)), and a liquid aerosol (e.g., DOP).

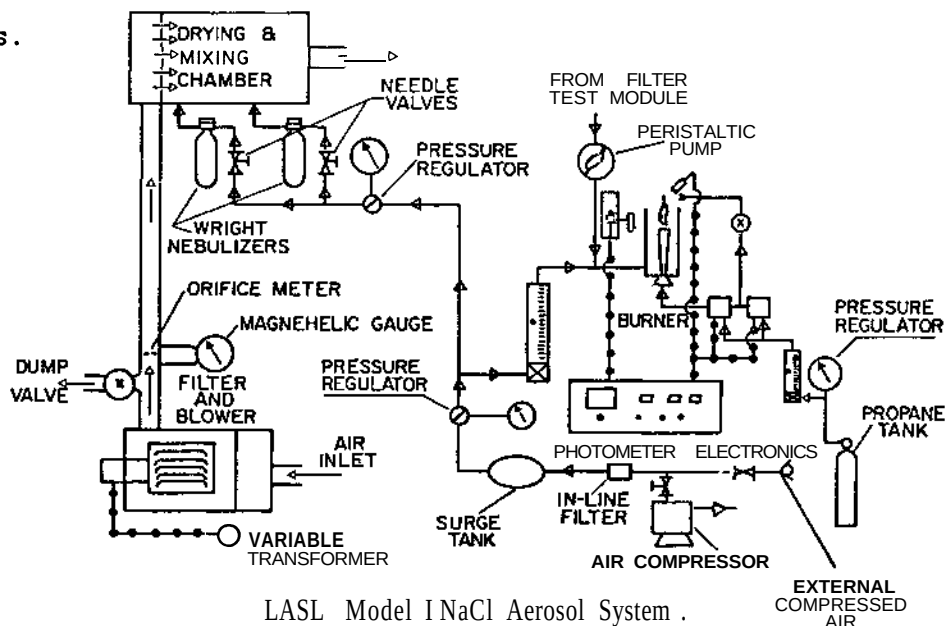
In order to study respirator particulate filter testing methodology, NIOSH conducted a multiphase investigation using the present respirator test aerosols and the proposed aerosols. Phase I of this investigation compared the filter penetration of four challenge aerosols (NaCl , DOP, SiO_2 , and PbO) with a variety of filter media. Phase II considered the effect of humidity and charge on the penetration of NaCl aerosol through particulate filter media. Phase III studied the long term effect of humidity on the change in NaCl penetration with a variety of particulate filter media. All filters used in this study were NIOSH approved under their respective test methodology in the current 30 CFR 11 regulations.

METHOD

Phase I

In Phase I, four brands of respirator particulate filters (dust and mist (DM); dust, fume and mist (DFM); and/or high efficiency particulate

(HEPA)) were tested with NaCl aerosol at NIOSH facilities in Cincinnati, Ohio. NaCl aerosol was generated by a Los Alamos Scientific Laboratory (LASL)* Model 1 NaCl aerosol system (Figure 1) as described in LASL report LA-5805-PR (1974). The aerosol generating system, using dry filtered air, consisted of a nebulizer assembly, a dilution air blower and a dilution air mixing chamber. The relative humidity of the NaCl aerosol was controlled to 50%. The aerosol, which was generated by a Wright nebulizer, had a particle size of 0.6 to 0.8 micrometer (μm) aerodynamic mass median diameter (AMMD) and a geometric standard deviation of 2. The NaCl particle size was measured with a Particle Measuring System (PMS) ASAS-X laser spectrometer and a set of Frontier FE400 NaCl impactors. The NaCl aerosol concentration was 15 mg/m^3 as determined by gravimetric means and a Thermal Systems Incorporated (TSI) 3205A particulate mass monitor. The instantaneous NaCl penetration was detected by propane flame photometry, at 32 L/min flow through the filters.



* LASL is currently Los Alamos National Laboratory (LANL).

The same particulate filters were also tested at the NIOSH testing and certification facilities in Morgantown, West Virginia against silica dust, lead fume, and DOP according to the 30 CFR Part 11 testing criteria. The silica dust, lead fume, and DOP aerosols were generated by NIOSH test systems operated as prescribed in Sections 11.140-4, 11.140-5, 11.140-6, and 11.140-11 of 30 CFR 11.

The average silica dust concentration, determined both gravimetrically and with the TSI mass monitor, was 56.4 mg/m^3 , while its count median diameter (CMD), measured with the laser spectrometer, was $0.36 \text{ }\mu\text{m}$. The silica dust penetrations were determined gravimetrically. The average lead fume concentration, also determined gravimetrically and with the TSI mass monitor, was 19.1 mg/m^3 , while its laser-measured CMD was $0.23 \text{ }\mu\text{m}$. The lead fume penetrations were determined gravimetrically.

The DOP test system was manufactured by Air Techniques Incorporated and generated a $0.3 \text{ }\mu\text{m}$ geometric mean diameter aerosol with a 1.2 geometric standard deviation (as determined by the manufacturer) at a gravimetrically determined, average concentration of 97 mg/m^3 . The DOP penetration was measured by light scattering photometry, at 42.5 L/min flow through the filters.

Phase II

LASL concluded from its interlaboratory reproducibility study, LA-7317-PR (1978), that there is significant variation in NaCl penetration from

laboratory to laboratory for a variety of filter media. For this reason, the effect of humidity and charge on the particulate filter penetration of NaCl aerosol was studied in Phase II. The LASL NaCl system was modified to control the humidity and aerosol charge while the particulate filter penetration of the NaCl aerosol was studied. The LASL NaCl system was modified to control humidity by adding either water vapor or dry filtered air to the aerosol generation system (Figure 1). The humidity in the test chamber was monitored by a Hydrocon I humidity controller. A TSI 3054 Kr-85 was used to help reduce the electrical charge on the NaCl aerosol particles. A Berkeley Controls piezoelectric cascade impactor was used to measure NaCl aerosol size. A representative assortment of particulate filters were tested at three humidities, 10%, 50%, and 90%, both with and without charge conditioning of the NaCl aerosol.

Phase III

The Phase III portion of this study evaluated the long-term (approximately 1 hour) change in NaCl penetration through several types of particulate filters at 10%, 50%, and 90% relative humidity. This portion was studied to help determine how filter loading and relative humidity affect filter penetration.

RESULTS AND DISCUSSION

Phase I Comparison of Particulate Filter Challenge Agents

Tables I-IV list the silica dust (SiO_2), lead fume (PbO), and DOP penetration test results as well as the NaCl (50% relative humidity) penetration results. The penetration of silica dust (Table I) for all filter types ranged from 0.06% for high efficiency particulate (HEPA) filters to 0.07% for dust and mist (DM) filters. These results indicate

TABLE I
Silica Dust (SiO_2) Penetration Data (32 Lpm)

Filter Manufacturer	Filter Type Description	Number Filter Pairs Tested	Mean SiO_2 Concentration	Arithmetic *Mean SiO_2 Penetration (90 Minutes)	Arithmetic Mean (Standard Deviation) Percent Penetration
A	Dust and Mist; Resin Impregnated	10	56.3 mg/m^3	0.12 mg	0.07% (0.03)
A	High Efficiency; Mechanical	10	56.0 mg/m^3	0.12 mg	0.07% (0.03)
B	Dust and Mist; Resin Impregnated	10	55.7 mg/m^3	0.12 mg	0.07% (0.03)
B	Dust, Fume, and Mist; Resin Impregnated and Mechanical	10	56.7 mg/m^3	0.10 mg	0.06% (0)
C	Dust, Fume, and Mist; Resin Impregnated and Mechanical	10	57.7 mg/m^3	0.12 mg	0.07% (0.04)
D	High Efficiency; Mechanical	10	56.0 mg/m^3	0.10 mg	0.06% (0)

* Minimum detectable limit is 0.10 mg

that the silica dust (SiO_2) test is not a sensitive indicator of the relative efficiency of respirator particulate filters. This is obvious since the HEPA and DM filters showed basically the same amount of

penetration. This "insensitivity" is probably due to the large size of the SiO_2 particulates. The lead fume (PbO) test (Table II) seems to be somewhat more differentiating than the SiO_2 test based upon the particulate filter efficiency. For the PbO test, the DM filters show a

TABLE II
Lead Fume (PbO) Penetration Data (32 Lpm)

Filter Manufacturer	Description Filter Type	Number Filter Pairs Tested	Mean PbO Concentration	*Mean PbO Penetration (312 Minutes)	Mean (Standard Deviation) Percent Penetration
A	(Dust and Mist)	10	19.1 mg/m^3	27.0 mg	14.2% (2.1)
A	(High Efficiency)	10	18.5 mg/m^3	0.27 mg	0.14% (0.27)
B	(Dust and Mist)	9	20.7 mg/m^3	18.2 mg	8.8% (1.5)
B	(Dust, Fume, and Mist)	10	19.7 mg/m^3	0.38 mg	0.19% (0.10)
C	(Dust, Fume, and Mist)	10	20.7 mg/m^3	0.19 mg	0.09% (0.05)
D	(High Efficiency)	10	18.1 mg/m^3	1.14 mg	0.63% (1.19)

* Minimum detectable limit is 0.10 mg

higher degree of penetration (8.8% to 14.2%) than either the DFM (0.09% to 0.19%) or the HEPA filters (0.14% to 0.63%). Although the DFM filters appear to be more efficient than the HEPA filters, this result is not statistically different. This anomaly may result from the PbO fume and SiO_2 aerosols being difficult to generate and maintain -at a stable mass concentration and particle size for the length of the respective test runs (90 minutes for SiO_2 and 312 minutes for PbO). Furthermore, the instantaneous breakthrough of SiO_2 and PbO aerosols cannot be determined at any one point in time during the tests. These results suggest that a smaller, more penetrating aerosol, which would be more discriminating, would be desirable for future routine respirator particulate filter testing.

The NaCl penetration results at 50% relative humidity are presented in Table III. The penetration results ranged from 0.03% (minimum detectable limit) for HEPA filters to 1.07% for DF&M to 6.70% for DM filters. By comparing percent penetrations, the polydispersed NaCl aerosol appears to be similar to PbO in its ability to measure filter efficiency of DM and DFM filters. At 50% relative humidity, the mean instantaneous NaCl penetrations for DM filters were 2.64% and 6.70% and for DFM filters were 1.07% and 0.30%. The measurement of penetration less than 0.03% for HEPA filters was not possible with the NaCl detection system employed. A more

TABLE III
NaCl (50% RH) Penetration Data (32 Lpm, without charge neutralizer)

<u>Filter Manufacturer</u>	<u>Description Filter Type</u>	<u>Number Filters Tested</u>	<u>NaCl Concentration</u>	<u>*Mean (Standard Deviation) Percent Penetration</u>
A	(Dust and Mist)	10	15.1 mg/m ³	6.70% (1.45)
A	(High Efficiency)	10	15.0 mg/m ³	0.03% (0)
B	(Dust and Mist)	10	14.8 mg/m ³	2.64% (0.28)
B	(Dust, Fume, and Mist)	10	14.8 mg/m ³	0.30% (0.06)
C	(Dust, Fume, and Mist)	10	14.7 mg/m ³	1.07% (0.43)
D	(High Efficiency)	10	14.7 mg/m ³	0.03% (0)

* Minimum detectable limit is 0.10 mg

sophisticated detection system, including the possible use of hydrogen flame instead of propane flame photometry, and a more refined electronics package, might detect much lower NaCl penetrations than 0.03%. However, the NaCl aerosol is easy to generate and the detector response for NaCl is much faster.

The DOP penetration (Table IV) ranged from 0.001% for high efficiency filters to 88.1% for dust and mist filters. The initial penetration of DOP for DM filters started at about 3% and reached 88% after about five minutes. This rapid increase in DOP penetration results from degradation of the filter media and was noted for some DFM filters as well. Thus, the DOP method appears to be the most sensitive, reliable, and rapid measure of particulate filter efficiency of all the test aerosols.

TABLE IV
DOP Penetration Data (42.5 Lpm)

Filter Manufacturer	Description Filter Type	Number Filters Tested	NaCl Concentration	*Mean (Standard Deviation) Percent Penetration
A	(Dust and Mist)	10	96.5 mg/m ³	88.1% (0.6)
A	(High Efficiency)	10	98.1 mg/m ³	0.001% (0.001)
B	(Dust and Mist)	10	96.5 mg/m ³	87.3% (1.0)
B	(Dust, Fume, and Mist)	10	97.8 mg/m ³	69.2% (4.4)
C	(Dust, Fume, and Mist)	10	97.8 mg/m ³	5.39% (1.46)
D	(High Efficiency)	10	98.1 mg/m ³	0.009% (0.013)

* Minimum detectable limit is 0.001%

Aerosol research (Lee, 1980) has shown that the 0.3 μm size of the DOP aerosol is in the range of particle sizes for maximum filter penetration. At this critical particle size, larger aerosol particles are removed by **impaction** (the process in which the inertia of particles in an **airstream** flowing around a respirator filter fiber causes the particles to strike and adhere to the **fiber**), while smaller particles are removed by diffusion (the net movement of particles due to **Brownian**

motion when a concentration gradient exists). At this crossover point (approximately 0.3 μm) for the different filtering mechanisms the minimum efficiency or maximum penetration should be observed.

In addition, DOP is also an excellent indicator of filter "degradation," i.e., decreased filter efficiency with time as measured by increased aerosol penetration. NIOSH is concerned that certain respirator particulate filters degrade under typical use and storage conditions. For example, Phase II results show resin impregnated (electrostatic) filter media may degrade in laboratory simulated humid-use conditions. Furthermore, NIOSH studies have shown significant degradation of electrostatic filter media in coke oven (Smith, 1979) and pesticide environments (Kennedy, 1983). Also, LASL has shown significant degradation for certain types of electrostatic filters after storage in humid environments (LA-5805-PR, 1974 and LA-6386-PR, 1974). The DOP data of Phase I indicate these same "degradable" electrostatic filters are adversely affected by the DOP oil mist and allow very high percent penetration of DOP aerosol (69.2% to 88.1%). This degradation phenomenon may be similar to the effects of coke oven and pesticide particulates and vapors on respirator particulate filters. Hence, for laboratory evaluation, a test using an oil based aerosol for measuring the efficiency and degradability of respirator particulate filters seems appropriate.

Although studies have shown that phthalate esters may cause teratogenic (Singh, 1972), and mutagenic (Singh, 1974) effects in laboratory animals,

the DOP aerosol apparatus is an enclosed system, thereby minimizing exposure. Also, potential exposure can be further reduced by using additional control measures such as ventilation and by the use of protective equipment. Furthermore, a less toxic liquid aerosol could be generated using oleic acid, mineral oil, corn oil, or silicone oil (NIOSH, 1983).

Phase II Effect of Humidity and Charge on NaCl Penetration

Table V and Figures 2-5 show that there is a significant effect on filter penetration due to charge and humidity. Table V lists the percent penetration of the NaCl aerosol for four brands of filters at 10%, 50%, and 90% relative humidity, with and without charge conditioning. The

TABLE V
NaCl Penetration Data with Humidity and
Charge Control (32 Lpm, 15 mg/m³)

Filter	Number Filters Tested	Relative Humidity	Mean (Standard Deviation) Percent Penetration Without Charge Neutralizer	Mean (Standard Deviation) Percent Penetration With Charge Neutralizer
A (Dust and Mist)	10	100%	16.9% (1.4)	15.0% (1.05)
	10	50%	6.7% (1.5)	8.4% (1.6)
	10	90%	6.1% (1.0)	6.5% (1.1)
B (Dust, Fume, and Mist)	10	100%	0.39 (0.06)	0.78 (0.12)
	10	50%	0.30 (0.06)	0.51 (0.10)
	10	90%	0.24 (0.12)	0.27 (0.13)
C (Dust, Fume, and Mist)	10	100%	11.42 (0.59)	1.60 (0.58)
	10	50%	11.07 (0.43)	1.16 (0.47)
	10	90%	1.00 (0.30)	0.95 (0.27)
D (Dust and Mist)	10	100%	112.2% (0.33)	2.24% (0.90)
	10	50%	1.10% (0.52)	1.40% (0.65)
	10	90%	0.75% (0.18)	1.12% (0.32)

corresponding plots are given as Figures 2-5, and show that for each filter, there is a decrease in NaCl penetration with increased humidity. This lowering of penetration with increasing humidity is probably the result of increased NaCl particle size with increasing humidity due to particle agglomeration and hydration. A Berkeley Controls piezoelectric cascade impactor was used to verify the larger NaCl aerosol size with increasing humidity. Although this instrument indicated a relative increase in particle size, no absolute determination was made.

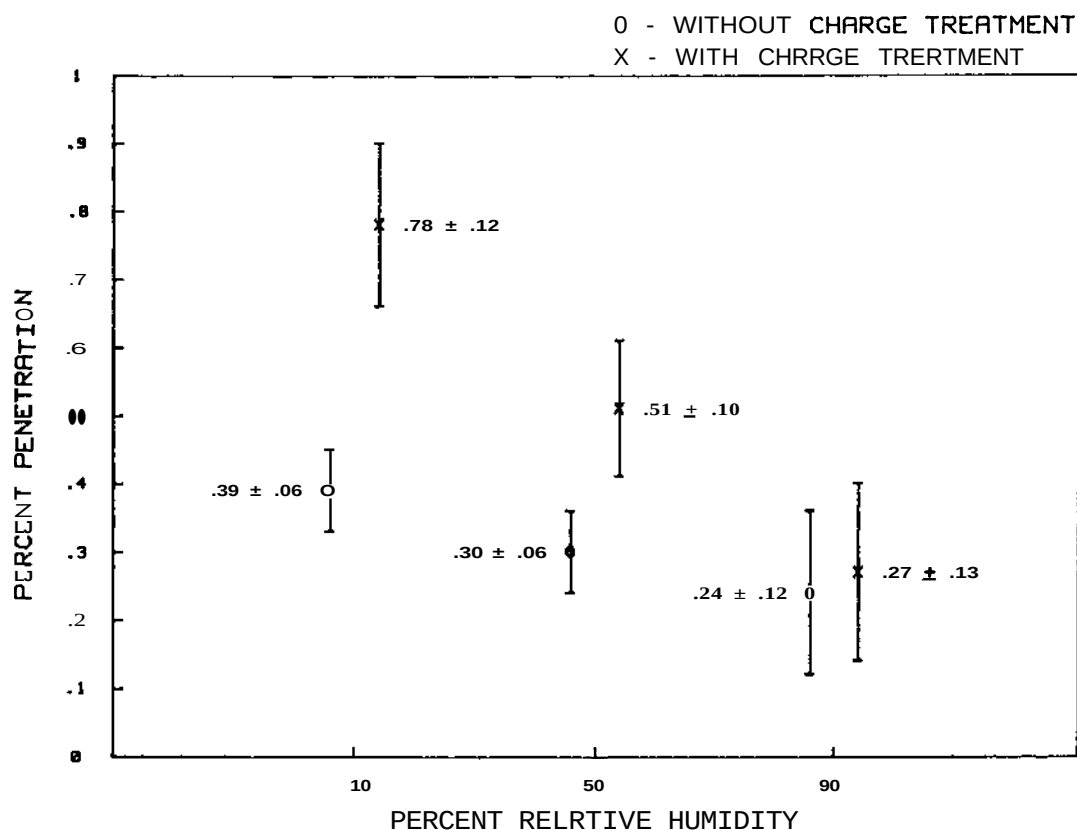


FIGURE 2
NaCl PENETRATION (MEAN ± 1 Std. Dev.) vs RELTIVE HUMIDITY FOR B(DFM)

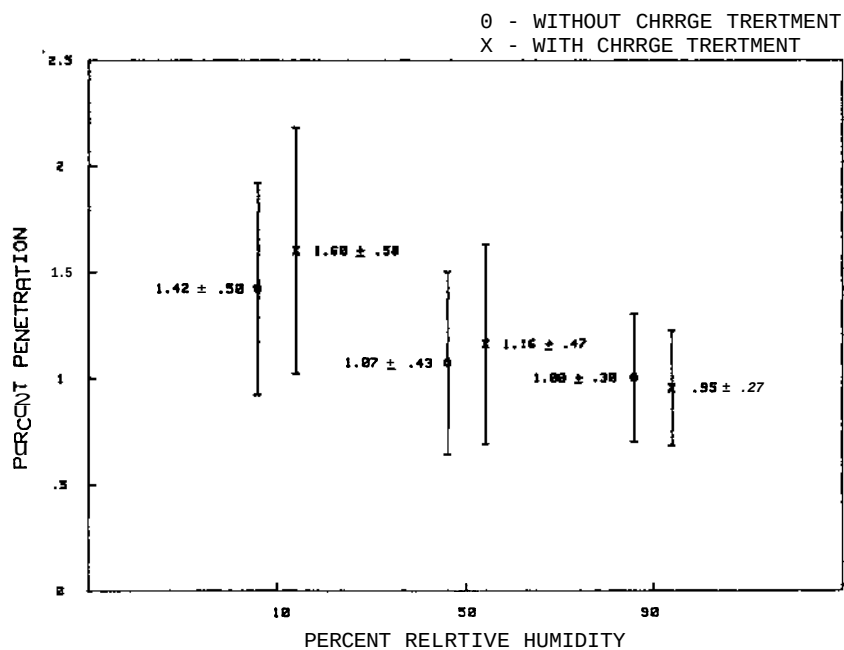


FIGURE 3
NaCl PENETRATION (MEAN ± 1 Std. Dev.) vs RELATIVE HUMIDITY FOR C(DFM)

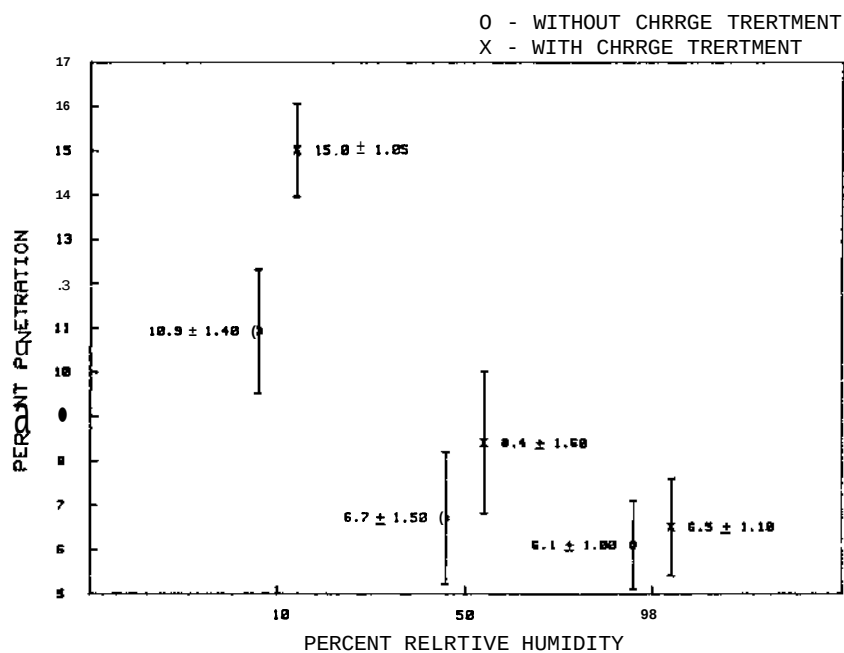


FIGURE 4
NaCl PENETRATION (MEAN ± 1 Std. Dev.) vs RELATIVE HUMIDITY FOR A(DH)

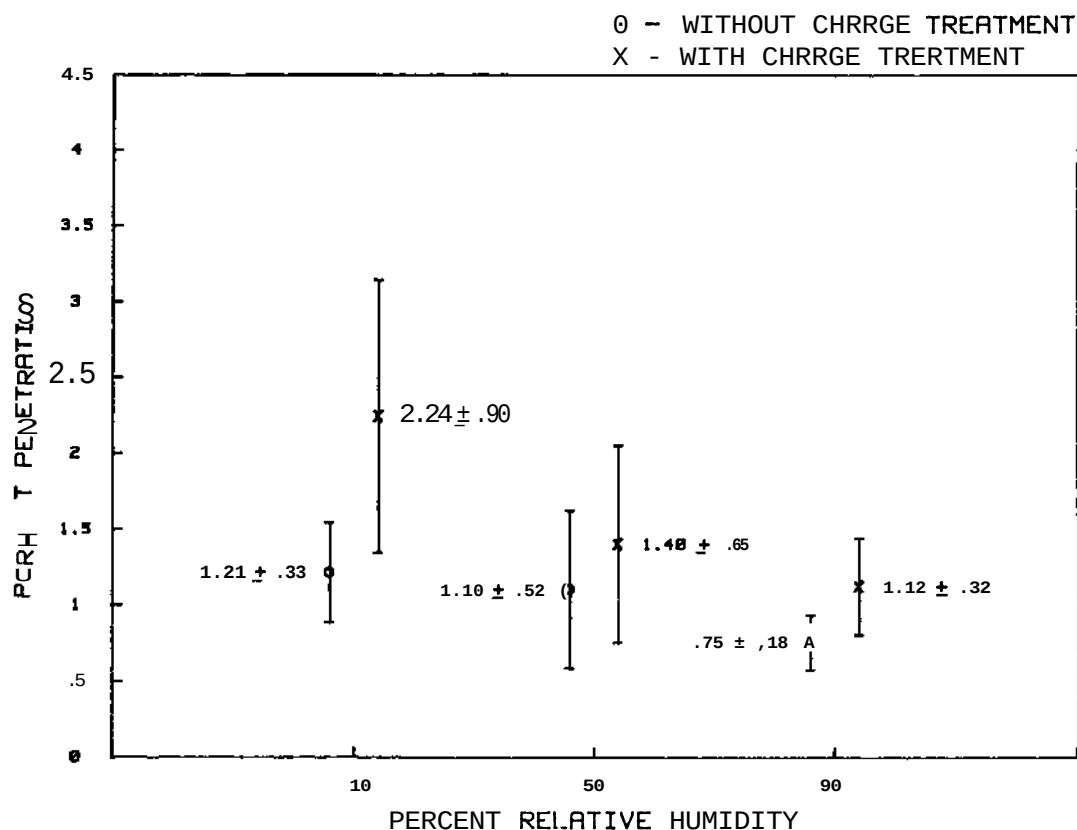


FIGURE 5
NaCl PENETRATION (MEAN ± 1 Std. Dev.) vs RELATIVE HUMIDITY FOR D(DM)

Table V shows there are significantly higher NaCl penetrations when the aerosol charge is reduced by conditioning with a TSI 3054 Kr-85 radioactive source (at 10%, 50% relative humidity). However, the relative difference in penetration between charged and uncharged aerosols decreases with increasing humidity. This decreasing difference in penetration with increasing humidity probably results from greater charge accumulating on NaCl particles when they are generated at lower humidities. The greater the charge, the more readily the particles are removed, especially by electrostatic filter media. At higher humidities, there is less particle charge and hence a smaller effect due to charge

neutralization. The humidity and charge phenomena help explain the differences in NaCl penetration between laboratories as reported by LASL (LA-7317-PR, 1978). Figures 2-5 graphically show the effect of humidity and charge on NaCl penetration. Based on these observations, humidity and charge should be controlled for all NaCl aerosol tests, including filter penetration and respirator quantitative fit-testing.

Phase III Long Term NaCl Penetration

The results of Phase III, although limited to only one filter per run, indicate the possible "degradation" effect of humidity on electrostatic filter media. Table VI shows the results of the 1 hour NaCl penetration through these filters at 10%, 50% and 90% relative humidity. As seen

TABLE VI

One-Hour NaCl Penetration
(15 mg/m³, 32 Lpm, without charge neutralizer)

Relative Humidity	Filter	Filter Type	Initial	Final	% Change
90%	A	DM	7.0%	24.0%	+242.9%
	B	DFM	0.15%	0.36%	+140.0%
	C	DFM	1.0%	0.9%	-10.0%
	D	DM	0.9%	6.4%	+611.1%
	E #1	DM	0.80%	2.75%	+243.8%
	E #2	DM	1.0%	3.5%	+250.0%
50%	A	DM	10.0%	27.0%	+170.0%
	B	DFM	0.4%	0.3%	-25.0%
	C	DFM	0.9%	0.7%	-22.2%
	D	DM	0.6%	0.6%	0.0%
	E #2	DM	2.4%	4.7%	+95.8%
10%	A	DM	10.0%	17.0%	+70.0%
	B	DFM			
	C	DFM	1.0%	0.3%	-70.0%
	D	DM	—		
	E #2	DM	3.3%	3.3%	0.0%

from a comparison of the initial to final penetration (percent change in penetration) at 90% relative humidity, all filters, except one, had a substantially higher final penetration overtime. Although some change in penetration was noted at 50% and 10% relative humidities, the increase in penetration overtime was not as great as that found at 90% relative humidity. That is, there appears to be much higher final NaCl penetration overtime for all "degradable" filters at 90% relative humidity than at lower humidities. Although more simulated, humidity testing is required to verify the degradation effect, there is enough evidence to indicate that filter degradation is an important problem that should be addressed in performance testing programs.

CONCLUSIONS

In conclusion, this study indicates that the SiO_2 test is not a sensitive indicator of the relative efficiency of respirator particulate filters. The SiO_2 penetration results were almost identical for all types of filter media (DM, DFM and HEPA). This insensitivity is partially due to the relatively large particle size of the SiO_2 aerosol. The PbO and polydispersed NaCl aerosols are reliable for distinguishing between filter types based upon the observed filter efficiencies. However, the NaCl system has more versatility than PbO and SiO_2 because instantaneous penetrations can be measured and because the NaCl aerosol concentration is more stable and easier to generate. Also, the NaCl aerosol size and charge, as well as the relative humidity of the challenge environment are easy to control.

This study showed the DOP aerosol to be the most severe and sensitive test for respirator particulate filter penetration. This is probably due to the fact that the DOP aerosol's size is in the most penetrating range (approximately $0.3\ \mu\text{m}$) and because DOP is a liquid aerosol which has a very degrading effect on some particulate filter media, especially the electrostatic type.

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