Summary

Four brands of respirator particulate filters were tested during 1978 for penetration using monodisperse di-octyl phthalate (MDOP), silica dust, lead fume, and sodium chloride aerosol. Results have shown that MDOP would be a reliable aerosol for measuring the efficiency and degradability of respirator particulate filters. A recommendation has been made to control humidity and charge for all NaCl aerosol use.

COMPARISON OF RESPIRATOR PARTICULATE FILTER TEST METHODS

by

Laurence D. Reed, David L. Smith, Thomas C. Mollman, I. Joel Frockt

U.S. Department of Health, Education and Welfare
National Institute for Occupational Safety and Health
Division of Physical Sciences and Engineering
Control Technology Research Branch
Robert A. Taft Laboratories
4676 Columbia Parkway
Cincinnati, Ohio 45226
August, 1980

NIOSH 00124130

REPORT DOCUMENTATION PAGE

1. REPORT NO. a

2. NA

3. Recipient's Accession No. [illegible] NA 159335

4. Title and Subtitle
Comparison of Respirator Particulate Filter Test MEthods

5. Report Date
August 1980

6. NA

7. Author(s)
Reed, L. D., D. L. Smith, T. C. Mollman, and I. J. Frockt

8. Performing Organization Rept. No.
NA

9. Performing Organization Name and Address
Hazard Evaluation and Technical Assistance Branch, Division of Surveillance, Hazard Evaluation and Field Studies, NIOSH, Cincinnati, Ohio

10. Project/Task/Work Unit No.
NA

11. Contract(C) or Grant(G) No.
(C)
(G)

12. Sponsoring Organization Name and Address
Same as Above

13. Type of Report & Period Covered
Technical Report

14. NA

15. Supplementary Notes
NA

16. Abstract (Limit: 200 words)

Four respirator particulate filters were evaluated. Penetration of monodisperse-dioctyl-phthalate (117817) (MDOP), silica (7631869), dust, lead (7439921) fumes, and sodium-chloride (7647145) (NaCl) aerosol was measured by light scattering and propane flame photometry. The effects of humidity and charge on NaCl penetration was also studied. For the test chemicals, penetration ranged from 0.001 to 88.1 percent, 0.06 to 0.07 percent, 0.09 to 14.2 percent, and 0.03 to 6.7 percent, respectively, on high efficiency and on dust and mist filters. For each filter type a decrease in NaCl penetration with increasing humidity was noted. Significantly higher NaCl penetrations were also observed when the aerosol charge was neutralized. The authors conclude detection might improve with a more sophisticated system using hydrogen flame, instead of propane flame photometry, and more refined electronic equipment.

17. Document Analysis a. Descriptors

Filter-materials, Filteration, Pulmonary-protective-equipment, Mateirals-testing, Analytical-methods, Analytical-instruments, Analytical-processes

b. Identifiers/Open-Ended Terms 0

c. COSATI Field/Group

18. Availability Statement
Available to Public

19. Security Class (This Report)
NA

20. Security Class (This Page)

21. No. of Pages
26

22. Price

COMPARISON OF RESPIRATOR PARTICULATE FILTER TEST METHODS

INTRODUCTION

Currently, NIOSH tests and certifies respirator particulate 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, lead fume, and 0.3 μm di-octyl phthalate (DOP) as challenge agents. Aerosols which are being considered as alternatives to the above tests include sodium chloride (NaCl), polydisperse DOP, and monodisperse DOP.

During 1978 NIOSH conducted a multiphase investigation to study the presently used respirator test aerosols and the proposed aerosols. Phase I of this investigation compared the filter penetrations of four aerosols (NaCl, monodisperse DOP, silica dust, and lead fume) 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.

METHOD

In Phase I, four brands (three types per brand) of respirator particulate filters 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) and as prescribed in a LASL report[1]. The aerosol generating system, using dry filtered air, consisted of a nebulizer assembly, a dilution air blower and a dilution air mixing chamber. The aerosol, which was generated by a Wright nebulizer, has a particle size of 0.6 to 0.8 micrometer (μm) aerodynamic mass mediam diameter (AMMD) and a geometric standard deviation of 2. NaCl aerosol concentration was 15 mg/m^3 as determined by gravimetric means and a Thermal Systems Incorporated (TSI) 3205A particle mass monitor. NaCl particle size was measured with a Particle Measuring System (PMS) ASAS-X laser spectrometer and a set of Frontier FE400 NaCl impactors. NaCl penetration was detected by propane flame photometry, at 32 L/min flow through the filters.

The same four brands of particulate filters were tested at NIOSH testing and certification facilities in Morgantown, West Virginia against silica dust, lead fume, and monodisperse DOP (MDOP). The silica dust and lead fume aerosols were generated by NIOSH test systems operated as prescribed in Sections 11.140-4, 11.140-5, and 11.140-6 of 30 CFR 11. The MDOP system was manufactured by Air Techniques Incorporated and generated a 0.3 μm geometric mean diameter aerosol (1.2 geometric standard deviation) at a gravimetrically determined, average concentration of 97 mg/m^3. The MDOP penetration was measured by light scattering photometry, at 42.5 L/min flow through the filters.

A representative assortment of particulate filters (ten pairs each) were also tested in Morgantown against lead fume and silica dust at 32 Lpm. 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 μm. 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 μm. The lead fume penetrations were determined gravimetrically.

LASL has concluded from its interlaboratory reproducibility study[2] 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 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 charge neutralizer was used to help remove 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 (four brands, ten each) were tested at three humidities, 10%, 50%, and 90%, both with and without charge neutralization.

Finally, in Phase III, the long term (∿ 1 hour) change in NaCl penetration through several types of particulate filters at 10%, 50%, and 90% relative humidity was studied.

RESULTS

Comparison of Particulate Filter Challenge Agents (Phase I)

Tables I-IV list the silica dust, lead fume, NaCl (50% relative humidity) and the MDOP penetration results. The penetration of silica dust (Table I) for all filter types ranged from 0.06% for high efficiency filters to 0.07% for dust and mist filters. Lead fume penetrations (Table II) ranged from 0.09% for dust, fume, and mist filters to 14.2% for dust and mist filters. However, disregarding dust and mist filters, the range of penetration, 0.09% to 0.63%, is also very small. NaCl penetration (Table III), at 50% relative humidity, ranged from 0.03% (minimum detectable limit) for high efficiency filters to 6.70% for dust and mist filters. Finally, MDOP penetration (Table IV) ranged from 0.001% for high efficiency filters to 88.1% for dust and mist filters. The initial penetration of MDOP for dust and mist filters started at about 3% and reached 88% after five minutes. This rapid increase in MDOP penetration was noted for some dust, fume, and mist filters as well.

Effect of Humidity and Charge on NaCl Penetration (Phase II)

Table V and figures 2-5 show the effect of charge and humidity on NaCl penetration through an assortment of particulate filters. Table V lists percent penetration of NaCl aerosol through four types of filters at 10%, 50%, and 90% relative humidity, with and without charge neutralization. Figures 2 - 5 show the plot of NaCl penetration versus relative humidity, with and without charge neutralization, by manufacturer. For each manufacturer or filter type, there is a decrease in NaCl penetration with increasing humidity. In addition, for each filter type, NaCl penetration at 10% and 50% humidity is significantly greater when charge is neutralized with the TSI 3054 charge neutralizer. However, these differences decrease with increasing humidity, so that at 90% humidity there are no significant differences in penetration between charged and charge neutralized NaCl aerosol.

Long Term NaCl Penetration (Phase III)

Table VI and figures 6 and 7 show the results of the long-term (∿ 1 hour) change in NaCl penetration through an assortment of particulate filters at 10%, 50% and 90% relative humidity. Table VI shows the initial and final penetration, and percent change in penetration for each humidity and filter type. At 90% relative humidity, all filter types, except MSA (S), had substantially higher final, than initial penetration. At 50%

relative humidity, the final penetration through MSA (S), AO (R-56), and Willson (R-30) either remained unchanged or decreased, while Norton (7500-6A)'s and 3M (9910)'s increased. However, this increase was not as much as that found at 90% relative humidity. At 10% relative humidity, MSA (S)'s final penetration decreased, 3M (9910)'s remained unchanged, and Norton (7500-6A)'s increased, but again, not as much as at 50% and 90% relative humidity. Figures 6 and 7 show the one-hour change in NaCl for the six filter types at 90% relative humidity.

CONCLUSIONS

Comparison of Particulate Filter Challenge Aerosols (Phase I)

The results of Phase I indicate silica dust (SiO_2) is not a sensitive indicator of the relative efficiency of respirator particulate filters. The SiO_2 penetration varies from 0.06% for high efficiency filters to only 0.07% for dust and mist filters. This "insensitivity" may be due to the large size of the SiO_2 particles compared with NaCl, MDOP, and lead fume aerosol particles. The larger sized particles are, of course, more easily captured by the filter media. Except for the dust and mist filters, lead fume (PbO) also seems to be a fairly insensitive indicator of particulate filter efficiency. That is, there is little spread in mean lead fume penetration between dust, fume, and mist filters (0.09%, 0.19%) and high efficiency filters (0.14%, 0.63%). However, dust and mist filters show much higher penetration of PbO (8.8%, 14.2%). In addition to being insensitive indicators of relative filter efficiency, lead fume and silica dust are difficult to generate and maintain at stable mass concentration and particle size for the length of the respective test runs (90 minutes for silica dust and 312 minutes for lead fume). Furthermore, the instantaneous breakthrough of silica dust and lead fume is difficult to accurately determine at any one point in time during the tests.

NaCl aerosol, on the other hand, appears to be a more sensitive measure of filter efficiency for dust and mist, and fume filters, than either silica dust or lead fume. At 50% relative humidity the instantaneous NaCl penetrations, for dust and mist filters, were 2.64% and 6.70%, while the fume filters were 1.07% and 0.30%. However, measurement of penetration less than 0.03% for high efficiency filters was not possible using the detection system of our NaCl test apparatus. A more 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%. Also, NaCl aerosol is easy to generate and the detector response for NaCl is much faster than for either lead fume or silica dust.

MDOP appears to be the most sensitive and reliable measure of particulate filter efficiency of all the test aerosols. Rapid (less than 5 minutes) and reliable filter penetration was obtained for all filter types. These MDOP penetrations ranged from 0.001% for high efficiency filters to 88% for dust and mist filters. Also, aerosol research has shown that the 0.3 μm size of the MDOP aerosol is the optimal size for maximum filter penetration[3]. Larger aerosol particles are removed by impaction (the process in which the inertia of particles in an airstream flowing around a fiber causes them to strike the fiber), while smaller particles are removed by diffusion (the net movement of particles due to Brownian motion when a concentration gradient exists).

In addition to the worst case condition of maximum penetration, MDOP 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[4] and pesticide environments.[5] And LASL has shown significant degradation for certain types of electrostatic filters after storage in humid environments.[1,6] The MDOP data of Phase I suggest these same "degradable" electrostatic filters are adversely affected by the oil mist MDOP, allowing very high percent penetration of MDOP aerosol (69.2% to 88.1%). This degradation phenomonen is possibly 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 is recommended for measuring the efficiency and degradability of respirator particulate filters.

Although studies have shown that phthalate esters may cause teratogenic,[7] and mutagenic[8] effects in laboratory animals, the MDOP and PDOP aerosol apparatuses which might be used to measure filter efficiency are enclosed systems, thereby minimizing exposure. Also, exposure could be reduced even further by using additional control measures and protective equipment. Furthermore, a potentially less toxic aerosol might be generated using oleic acid, mineral oil, or silicone oil.

Effects of Humidity and Charge on NaCl Penetration (Phase II)

The results of Phase II indicate there are significant differences in NaCl penetration due to charge and humidity for each manufacturer and filter type. Table V shows for each filter type there is a decrease in NaCl penetration with increasing humidity. The lower penetration with increasing humidity is probably the result of increased NaCl particle size with increasing humidity. The increased aerosol size with humidity is due to particle agglomeration and particle hydration. A Berkeley Controls piezoelectric cascade impactor was used to verify the larger NaCl aerosol size with increasing humidity.

Table V also shows there are significantly higher NaCl penetrations when the aerosol charge is neutralized, (at 10%, 50% relative humidity) but those differences decrease with increasing humidity (e.g., at 90% relative humidity). This decreasing difference in penetration probably results from greater charge mobility on NaCl particles when they are generated at lower humidities. Hence, the difference in penetration due to charge neutralization is greater at lower humidities because of the greater difference in electrical mobility between the neutralized and non-neutralized aerosol. Also, the more charge-mobile particles are more easily removed by electrostatic filter media. At higher humidities, there is less particle charge mobility and hence little effect due to charge neutralization. These effects might explain the differences in NaCl penetration between laboratories as reported by LASL.[2] Figures 2-5 graphically show the effect of humidity and charge mobility on NaCl penetration. In light of these observations, humidity and charge be controlled for all NaCl aerosol use, including filter penetration and respirator quantitative fit-test apparatuses.

Long Term NaCl Penetration (Phase III)

The results of Phase III, although limited to only one filter per run, indicate the possible "degradation" effect of humidity on resin impregnated or electrostatic filter media. Table VI and figures 6 and 7 indicate there was much higher final NaCl penetration for all "degradable" filters at 90% relative humidity than at lower humidities. Although more simulated, humid-use testing is required to verify the degradation effect, there is enough evidence to indicate filter degradation is an important problem that should be recognized in performance testing programs.

REFERENCES

1. Los Alamos Scientific Laboratory Progress Report: LA-5805-PR, December 1974.

2. Los Alamos Scientific Laboratory Progress Report: LA-7317-PR, June 1978.

3. Lee, K.W., and Liu, B.Y.H. Liu, "On the Minimum Efficiency and the Most Penetrating Particle Size for Fibrous Filters", Air Pollution Control Association Journal, Volume 30, April 1980, pp 377-381.

4. Smith, D.L., et al., "The Efficiency of Respirator Filters in a Coke Oven Atmosphere," American Industrial Hygiene Association Journal, Volume 40, No. 12, December 1979, pp. 1030-1038.

5. Kennedy, E.R., and L.D. Reed, "Evaluation of Pesticide Respirator Cartridges," unpublished NIOSH report.

6. Los Alamos Scientific Laboratory Progress Report: LA-6386-PR, May 1974.

7. Singh, A.R., et al., "Teratogenicity of Phthalate Esters in Rats," Journal of Pharmaceutical Sciences, Volume 61, No. 1, January, 1972, pp. 51-55.

8. Singh, A.R., et al., "Mutagenic and Antifertility Sensitives of Mice to Di-2-ethylhexyl Phthalate (DEAP) and Dimethoxyethyl Phthalate (DMEP)," Toxicity and Applied Pharmacology, Volume 29, 1974, pp. 35-46.

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

Filter Manufacturer	Filter Type	Number Filter Pairs Tested	Mean SiO_2 Concentration	*Mean SiO_2 Penetration (90 minutes)	Mean (Standard Deviation) Percent Penetration
Norton	7500-6A (Dust and Mist)	10	56.3 mg/m^3	0.12 mg	0.07% (0.03)
AO	R-30 (Dust and Mist)	10	55.7 mg/m^3	0.12 mg	0.07% (0.03)
MSA	S (Dust, Fume, and Mist)	10	57.7 mg/m^3	0.12 mg	0.07% (0.04)
AO	R-56 (Dust, Fume, and Mist)	10	56.7 mg/m^3	0.10 mg	0.06% (0)
Willson	R-12 (High Efficiency)	10	56.0 mg/m^3	0.10 mg	0.06% (0)
Norton	7500-8 (High Efficiency)	10	56.0 mg/m^3	0.12 mg	0.07% (0.03)

* Minimum detectable limit is 0.10 mg

TABLE II. Lead Fume (Pb Oxide) Penetration Data (32 Lpm)

Filter Manufacturer	Filter Type	Number Filter Pairs Tested	Mean Pb Oxide Concentration	*Mean Pb Oxide Penetration (312 minutes)	Mean (Standard Deviation) Percent Penetration
Norton	7500-6A (Dust and Mist)	10	19.1 mg/m^3	27.0 mg	14.2% (2.1)
AO	R-30 (Dust and Mist)	9	20.7 mg/m^3	18.2 mg	8.8% (1.5)
MSA	S (Dust, Fume, and Mist)	10	20.7 mg/m^3	0.19 mg	0.09% (0.05)
AO	R-56 (Dust, Fume, and Mist)	10	19.7 mg/m^3	0.38 mg	0.19% (0.10)
Willson	R-12 (High Efficiency)	10	18.1 mg/m^3	1.14 mg	0.63% (1.19)
Norton	7500-8 (High Efficiency)	10	18.5 mg/m^3	0.27 mg	0.14% (0.27)

* Minimum detectable limit is 0.10 mg

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

Manufacturer	Filter Type	Number Filters Tested	NaCl Concentration	*Mean (Standard Deviation) Percent Penetration
Norton	7500-6A (Dust and Mist)	10	15.1 mg/m^3	6.70% (1.45)
AO	R-30 (Dust and Mist)	10	14.8 mg/m^3	2.64% (0.28)
MSA	S (Dust, Fume, and Mist)	10	14.7 mg/m^3	1.07% (0.43)
AO	R-56 (Dust, Fume, and Mist)	10	14.8 mg/m^3	0.30% (0.06)
Willson	R-12 (High Efficiency)	10	14.7 mg/m^3	0.03% (0)
Norton	7500-8 (High Efficiency)	10	15.0 mg/m^3	0.03% (0)

*Minimum detectable limit is 0.03%

TABLE IV. MDOP Penetration Data (42.5 Lpm)

Filter Manufacturer	Filter Type	Number Filters Tested	MDOP Concentration	*Mean (Standard Deviation) Percent Penetration
Norton	7600-6A (Dust and Mist)	10	96.5 mg/m^3	88.1% (0.6)
AO	R-30 (Dust and Mist)	10	96.5 mg/m^3	87.3% (1.0)
MSA	S (Dust, Fume, and Mist)	10	97.8 mg/m^3	5.39% (1.46)
AO	R-56 (Dust, Fume, and Mist)	10	97.8 mg/m^3	69.2% (4.4)
Willson	R-12 (High Efficiency)	10	98.1 mg/m^3	0.009% (0.013)
Norton	7500-8 (High Efficiency)	10	98.1 mg/m^3	0.001% (0.001)

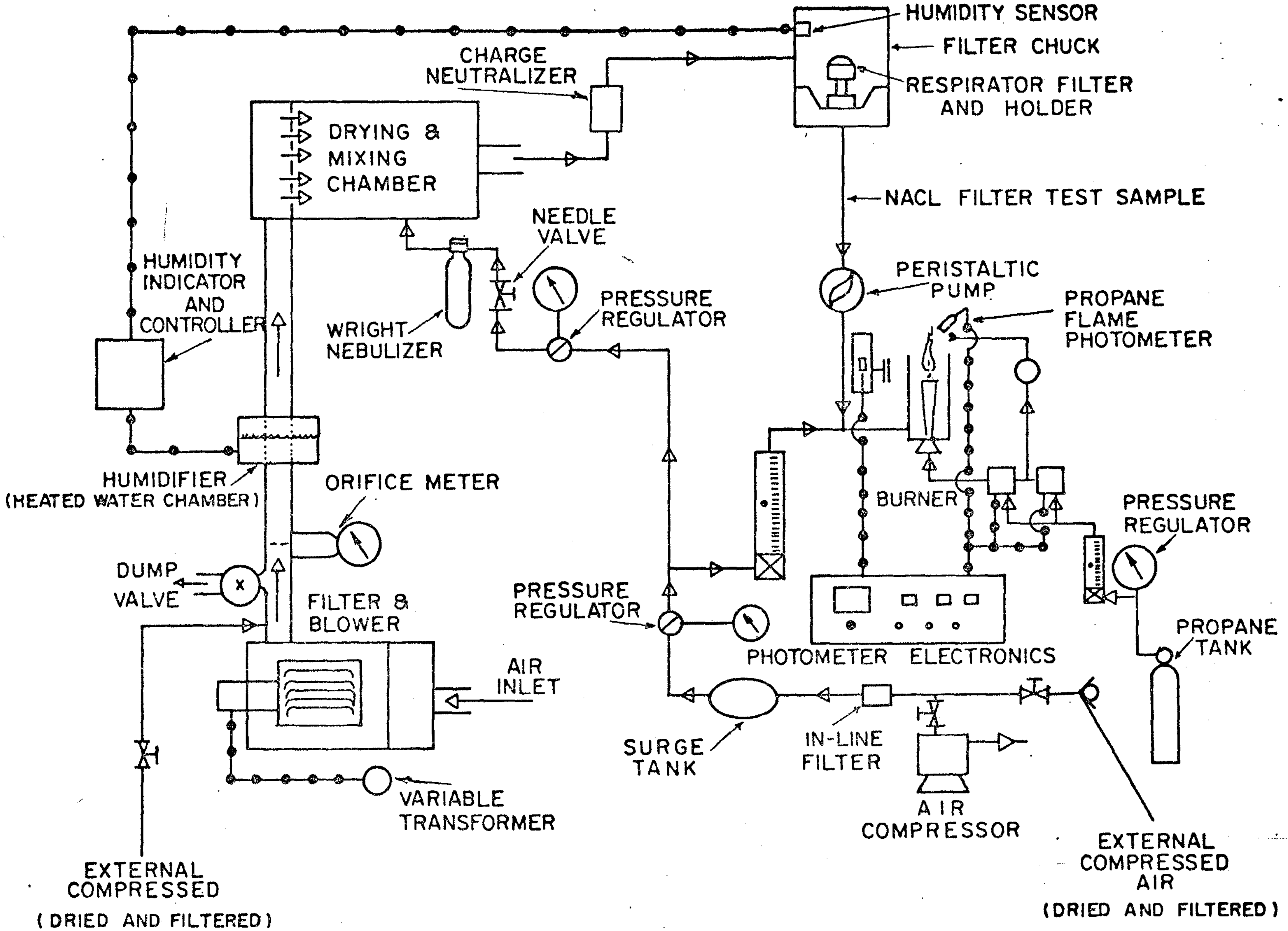
* Minimum detectable limit is 0.001%

TABLE V: NaCl Penetration Data with Humidity and Charge Control (32 Lpm, 15 mg/m^3)

Filter	Number Filters Tested	Relative Humidity	Mean (Standard Deviation) Percent Penetration Without Charge Neutralizer	Mean (Standard Deviation) Percent Penetration With Charge Neutralizer
AO, R-56 (Dust, Fume, and Mist)	10	10%	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)
MSA, S (Dust, Fume, and Mist)	10	10%	1.42 (0.50)	1.60 (0.58)
	10	50%	1.07 (0.43)	1.16 (0.47)
	10	90%	1.00 (0.30)	0.95 (0.27)
Norton, 7500-6A (Dust and Mist)	10	10%	10.9% (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)
Willson, R-30 (Dust and Mist)	10	10%	1.21% (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)

TABLE VI: One-Hour NaCl Penetration
(15 mg/m^3, 32 Lpm, without charge neutralizer)

Relative Humidity	Filter	Initial	Final	% Change
90%	MSA (S) (Dust, Fume, and Mist)	1.0%	0.9%	- 10.0%
	AO (R-56) (Dust, Fume, and Mist)	0.15%	0.36%	+140.0%
	Norton (7500-6A) (Dust and Mist)	7.0%	24.0%	+242.9%
	Willson (R-30) (Dust and Mist)	0.9%	6.4%	+611.1%
	3M 9900 (Dust and Mist)	0.80%	2.75%	+243.8%
	3M 9910 (Dust and Mist)	1.0%	3.5%	+250.0%
50%	MSA (S)	0.9%	0.7%	- 22.2%
	AO (R-56)	0.4%	0.3%	- 25.0%
	Norton (7500-6A)	10.0%	27.0%	+170.0%
	Willson (R-30)	0.6%	0.6%	0.0%
	3M 9910	2.4%	4.7%	+ 95.8%
10%	MSA (S)	1.0%	0.3%	- 70.0%
	AO (R-56)	--	--	--
	Norton (7500-6A)	10.0%	17.0%	+ 70.0%
	Willson (R-30)	--	--	--
	3M 9910	3.3%	3.3%	0.0%



Modified LASL Model 1 NaCl Aerosol System.

FIGURE 1

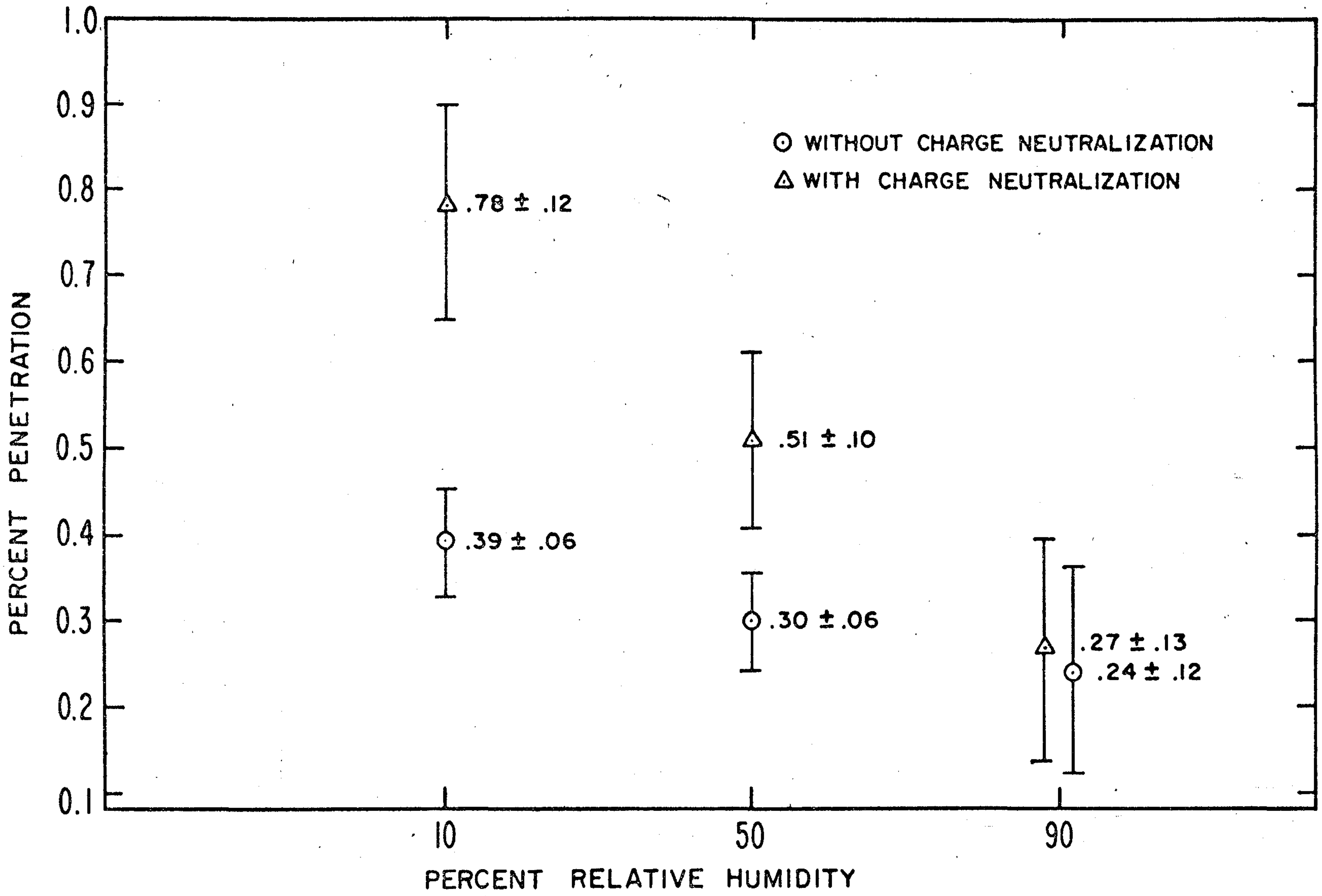


FIGURE 2
NaCl PENETRATION (MEAN ± 1 Std.Dev.) vs RELATIVE HUMIDITY FOR AO (R-56)

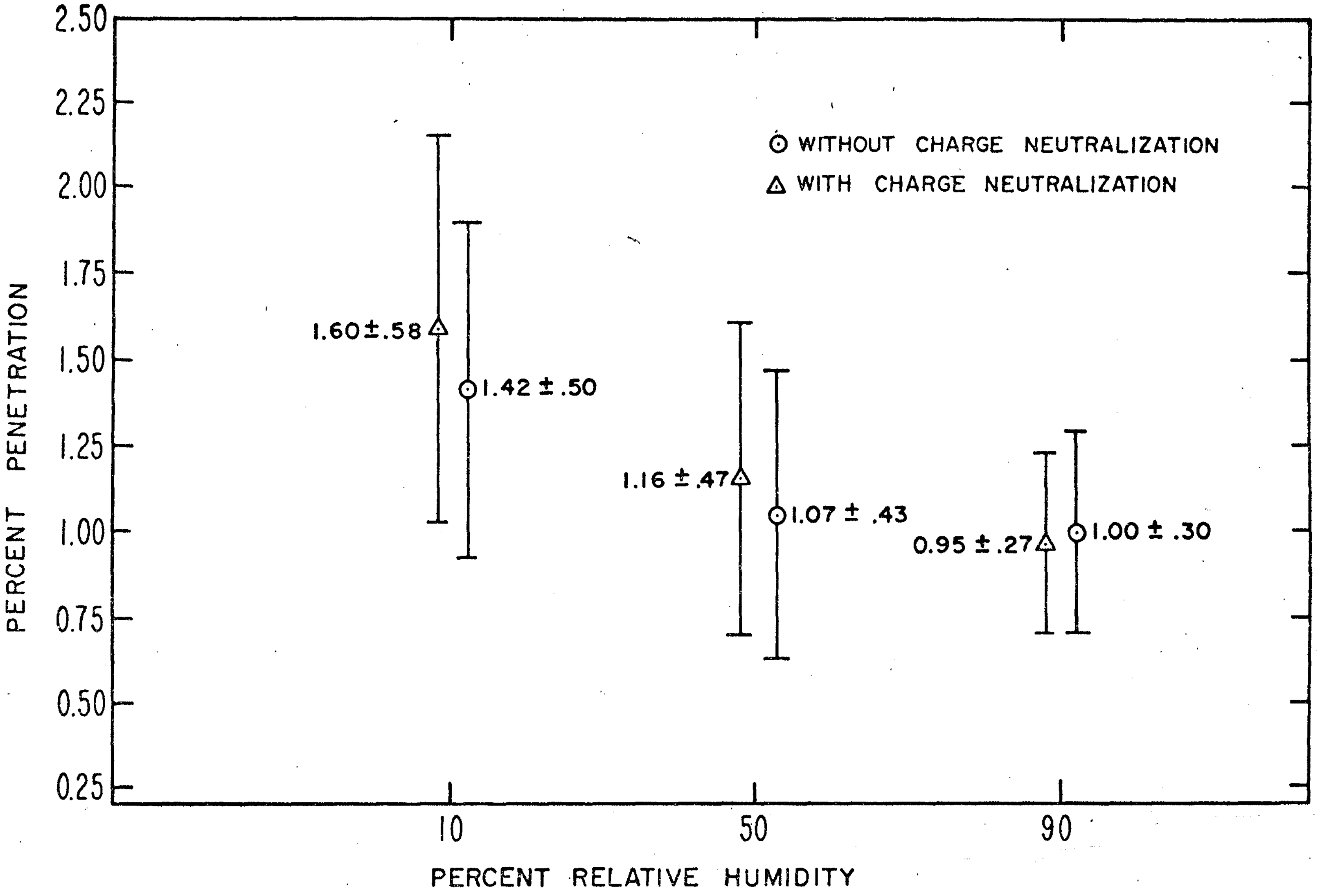


FIGURE 3
NaCl PENETRATION (MEAN ± 1 Std.Dev.) vs RELATIVE HUMIDITY FOR MSA(S)

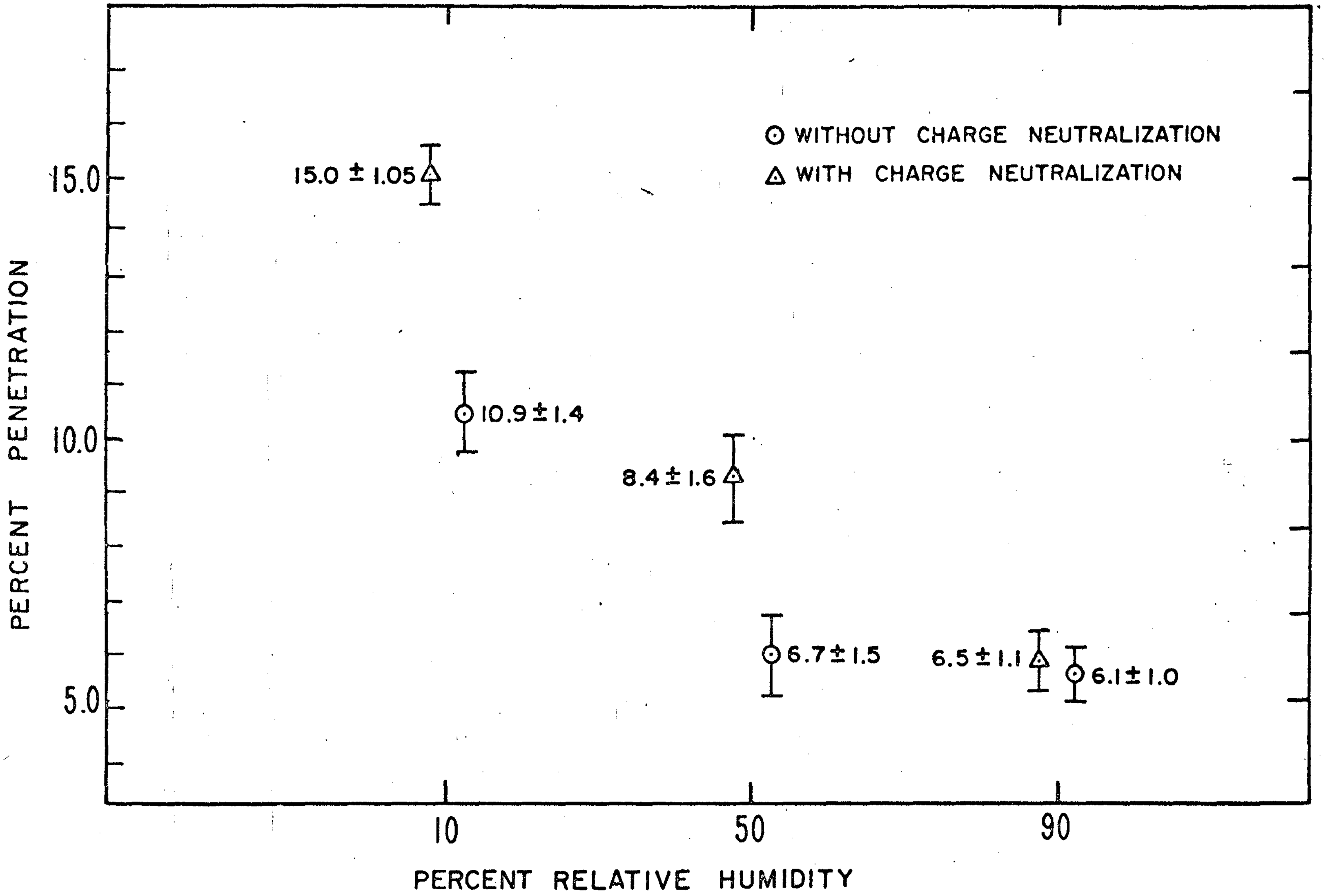


FIGURE 4
NaCl PENETRATION (MEAN $\pm$ 1 Std.Dev) VS RELATIVE HUMIDITY FOR NORTON (7500-6A)

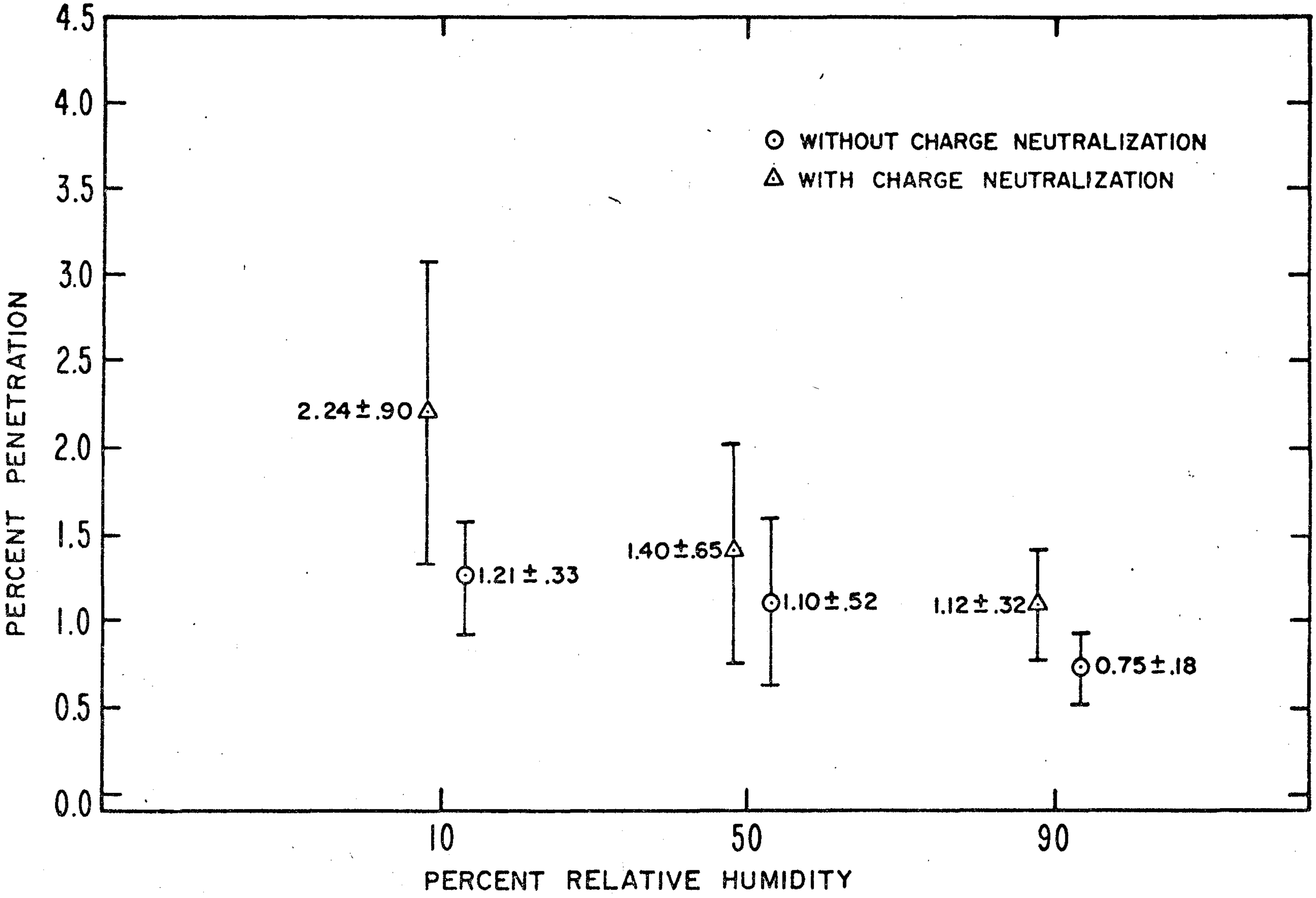


FIGURE 5
NaCl PENETRATION (MEAN ± 1 Std.Dev.) vs RELATIVE HUMIDITY FOR WILLSON (R-30)

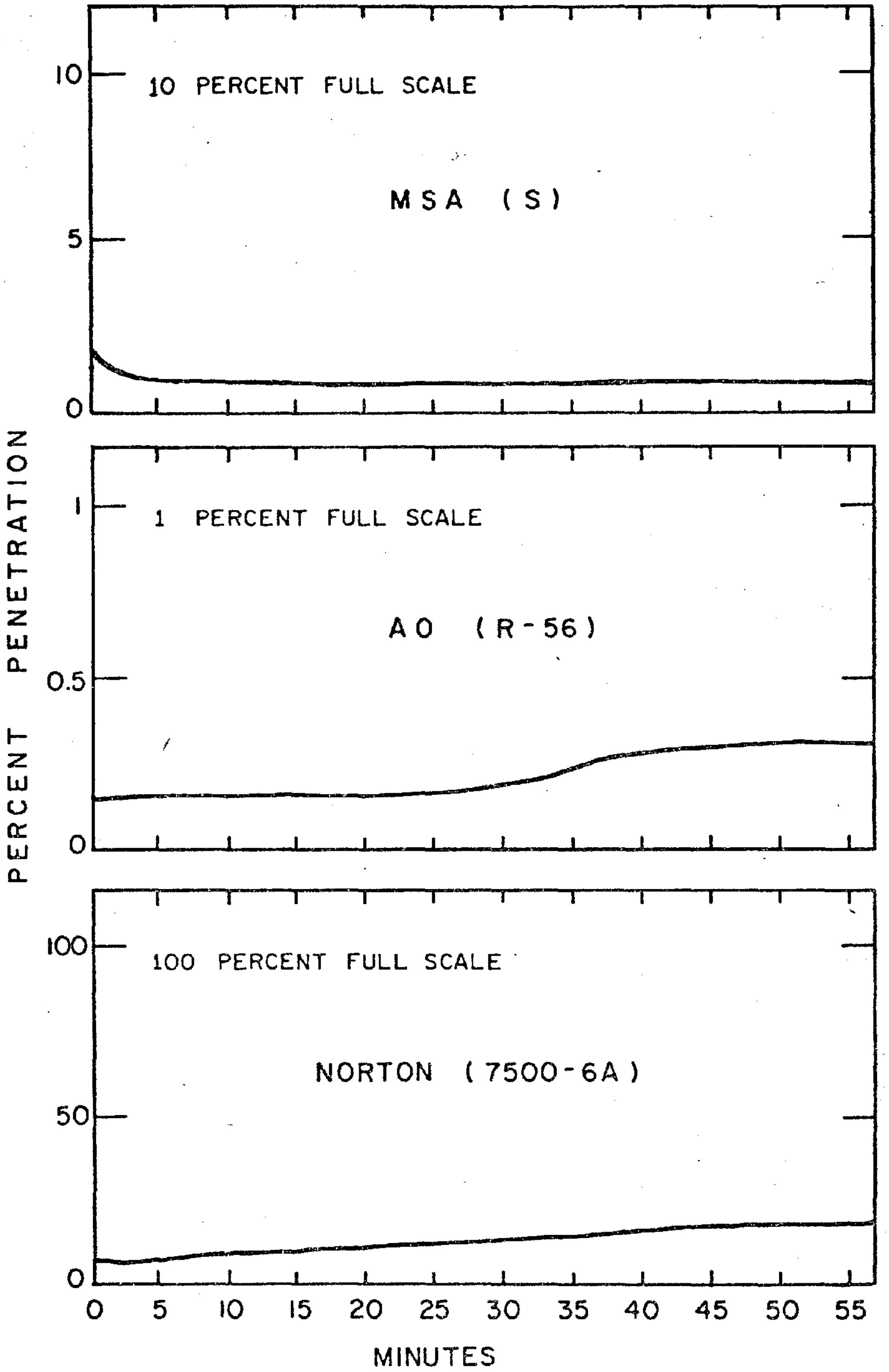


FIGURE 6

ONE HOUR NaCl PENETRATION AT 90% RH WITHOUT CHARGE NEUTRALIZATION

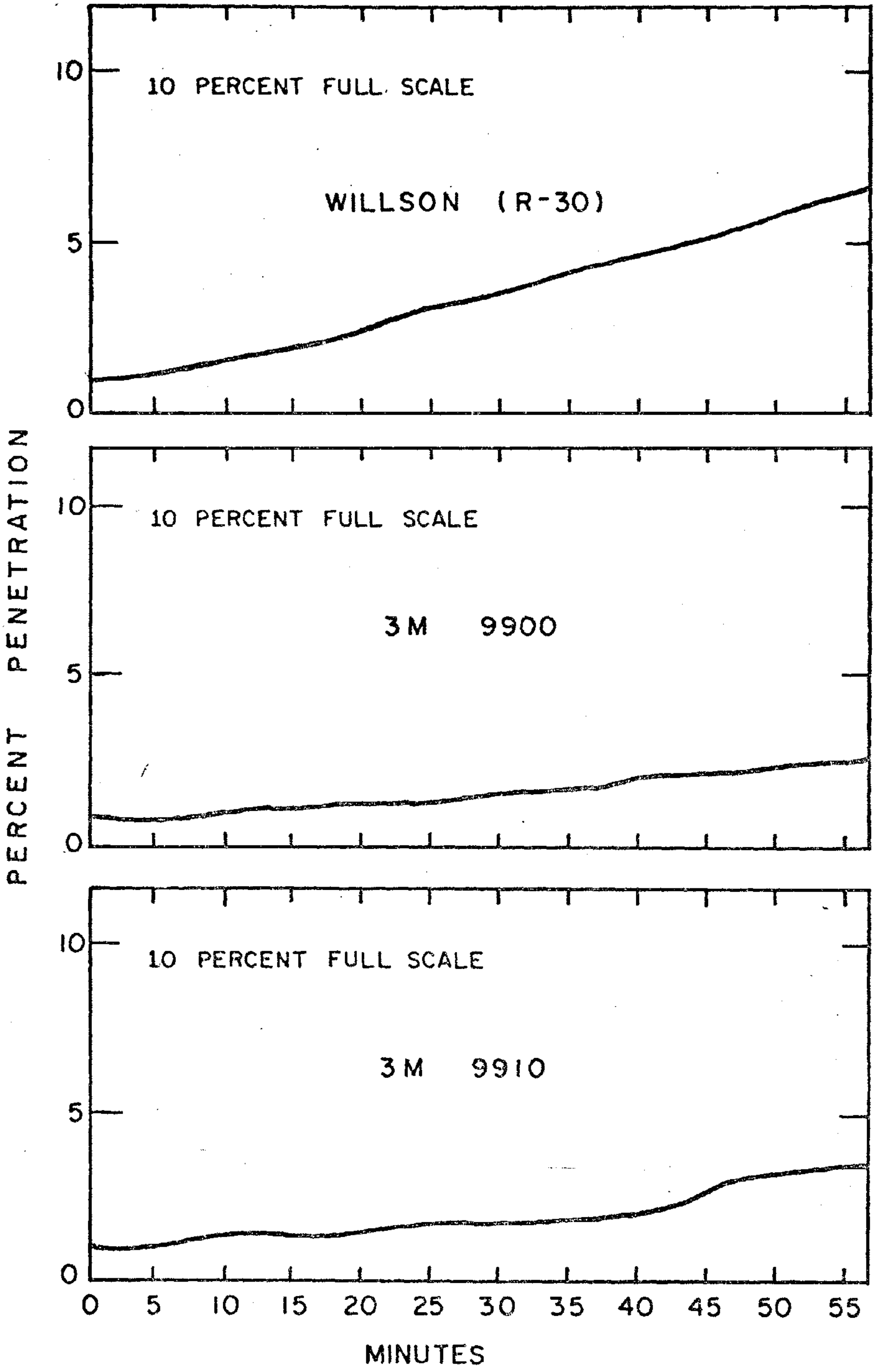


FIGURE 7

ONE HOUR NaCl PENETRATION AT 90% RH WITHOUT CHARGE NEUTRALIZATION