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**Development and Evaluation
of DOP Generators for Special Uses**

Robert L. Mielke

June 18, 1976

Research and Development Report

MOUND LABORATORY

Miamisburg, Ohio

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ABSTRACT

The Environmental Standards Group at Mound Laboratory uses "cold" DOP generators to produce background aerosol concentration for testing HEPA filters in clean-room operations. Two simple, portable, air-operated aerosol generators were built and tested to determine their performance.

The results show the particle size distribution is in the 0.3 to 0.8 μ m range and that 1/4 or 3/8 in. i.d. tubing to the nozzles should be used.

INTRODUCTION

Clean room calibration procedures¹ require the total scan test of HEPA (High Efficiency Particulate Air) filters using DOP (dioctyl-phthalate) aerosol. There are two methods to generate this DOP smoke concentration, hot and cold. (See Appendix.) The Environmental Standards Group at Mound Laboratory uses the cold generation method for this type of testing. Although commercial DOP generators are available, they do not entirely meet the special needs of the Environmental Standards Group. Two DOP generators were built and tested for particle size range and smoke concentration, and data were tabulated to determine their performance.

CONSTRUCTION OF DOP GENERATORS

The basic DOP generator design was taken from Sandia Process Standard 9956015,

FIGURE 1 - Brass atomizing nozzle used for both generators.

"Leak Testing, HEPA Filter Installations" Section 6.2, DOP Smoke Generators.

Two simple, air-operated aerosol generators were built, one (M1) having one nozzle with four jets and the other (M6) having six nozzles with four jets each and control valves so that from one to six nozzles could be operated at any one time. Both generators have pressure gauges for all nozzles. The nozzle shown in Figure 1 was used for both the M1 and M6 generators.

The M1 generator is shown in Figures 2-5. To increase the smoke generation, the pre-filter material and one of the perforated metal plates were removed from the M1 generator after the first set of tests. The modified generator was designated M1M.

The M6 generator is shown in Figure 6.

FIGURE 2 - One-nozzle generator M1 before modification.

FIGURE 3 - One-nozzle DOP generator.

FIGURE 4 - One-nozzle DOP generator with perforated plate.

FIGURE 5 - One-nozzle DOP generator with lid.

FIGURE 6 - Six-nozzle generator.

PERFORMANCE EVALUATIONS

The 24 x 24 x 5-7/8 in. HEPA filter used for evaluating the M1, M1M, and M6 generators was modified by cutting a section from the filter and installing a 3-3/8 in. diam x 3 in. deep cardboard cup, bottom down, with RTV sealant. A 1/4-in. hole was made in the center of the cup to allow for sampling of the DOP aerosol concentration (Figure 7). This filter was then installed in the 24 x 24 in. plenum box (Figure 8) for testing HEPA filters.

We used two types of particle sensing devices. One was a Rayco PC200 particle counter which had been modified at Sandia Laboratories² to include a dilution system (Figure 8) which, when activated, dilutes the sample going to the particle counter by a factor of 10, 100, or 1000 (i.e., 9, 99, or 999 parts clean air to one part sample air). The particle counter is able to differentiate the particulate matter which passes through the optical sensing chamber and provides both amount (number) and size (μm) data. The other device used was a Sinclair Phoenix Forward Scattering Aerosol, Dust and Smoke Photometer JM2000 (Figure 8). The aerosol sample passes through the optical sensing chamber of the photometer which measures the amount of light which has

FIGURE 7 - Modified HEPA filter.

FIGURE 8 - Test equipment with DOP generator.

been scattered by the particulate matter. This measurement is a level of the mass concentration and can be evaluated on the graph provided with the photometer (see Figure 9).

Data were also tabulated from tests of a two-nozzle DOP Generator (S2) which had been built by Sandia Laboratories, Manufacturing Research Division 2564. The tests performed are listed in Table 1.

RESULTS

The results of the tests are tabulated in Tables 2-5.

Table 2 shows the relationship between the M1 and M1M DOP generators. The DOP aerosol concentration increased approximately 35% after the perforated plate and prefilter material were removed from M1. However, even in the modified state, this generator does not approach the performance that was expected.

Table 3 shows the particle size distributions for all the DOP generators at 20 psi. As can be seen, the distribution is similar in most cases, with the highest

concentration at the $0.3 \mu\text{m}$ size level and decreasing as the particle size increases. However, in the M6-6 test the highest concentration occurred at the 0.5 and $0.6 \mu\text{m}$ levels. In all cases, at about the $1.2 \mu\text{m}$ level, the concentration began dropping drastically, and at $4.0 \mu\text{m}$ and above no particles were detectable. These results were acceptable for our use.

Table 4 shows the DOP aerosol concentration as determined by the photometer. When the data from the M1M generator and the M6 generator with one nozzle open are compared (Figure 10), it can be seen that the M6 generator produces approximately 500% more DOP concentration. This increase is due to the large inlet tubing size of the M6 generator compared to that of the M1M generator.

The data from the M6 generator with two nozzles open compare very closely with those from the Sandia-built two-nozzle generator (Figure 11).

The data from the M6 generator tests with one, two, three, and six nozzles open show that the DOP concentration levels go up as would be expected (Figure 12). These data correlate with the particle counter tests.

FIGURE 9 - Calibration of filter positions for aerosol photometer.

Table 1

TESTS PERFORMED TO DETERMINE DOP CONCENTRATION

Test	Pressure (psi)	Generators Tested			
		M1	M1M	M6	S2
0.5 μm and above ^a	5	X	X		X
	10	X	X		X
	15	X	X		X
	20	X	X	X ^b	X
	25	X	X		X
	30	X	X		
0.3 to 4.0 μm ^c	20	X	X	X ^d	X
Photometer	5		X	X ^e	X
	10		X	X ^e	X
	15		X	X ^e	X
	20		X	X ^e	X
	25		X	X ^e	X
	30		X	X ^e	X

^aParticles/ft³ of air/min equivalent.

^bTested with one, two, three, four, five, and six nozzles open.

^c0.3 to 4.0 μm particle distribution was tested per ft³ of air per min equivalent.

^dTested with one, three, and six nozzles open.

^eTested with one, two, three, and six nozzles open.

Table 2

DOP CONCENTRATIONS PRODUCED BY GENERATORS
M1 and M1M AT DIFFERENT PRESSURES

Pressure (psi)	DOP Concentration ^a from Generators	
	M1	M1M
5	0.4×10^7	0.58×10^7
10	1.1×10^7	1.44×10^7
15	1.5×10^7	1.92×10^7
20	1.8×10^7	2.35×10^7
25	2.1×10^7	2.69×10^7
30	2.3×10^7	2.98×10^7

^a Rounded off average number of particles 0.5 μm and above per ft³ of air per min over a 10-min period using the particle counter with the dilution set on 10^{-2} .

Table 3

PARTICLE SIZE CONCENTRATION DISTRIBUTION TESTS^a

Particle Size (μm)	Concentration Produced by Generators (particles/ft ³ /min)					
	<u>M1</u>	<u>M1M</u>	<u>M6-1^b</u>	<u>M6-3^c</u>	<u>M6-6^d</u>	<u>S2</u>
0.3	3.1×10^7	3.7×10^7	19×10^7	35×10^7	26×10^7	32×10^7
0.4	2.5×10^7	3.1×10^7	14×10^7	33×10^7	30×10^7	27×10^7
0.5	1.9×10^7	2.5×10^7	10×10^7	28×10^7	34×10^7	21×10^7
0.6	1.4×10^7	1.8×10^7	6.5×10^7	22×10^7	34×10^7	15×10^7
0.8	1.0×10^7	1.2×10^7	3.5×10^7	14×10^7	30×10^7	8.7×10^7
1.2	2.0×10^6	2.4×10^6	4.9×10^6	26×10^6	97×10^6	14×10^6
1.5	6.0×10^5	5.5×10^5	1.0×10^6	7.0×10^6	33×10^6	3.5×10^6
2.0	8.0×10^4	8.2×10^4	1.0×10^5	9.6×10^5	64×10^5	4.5×10^5
3.0	1.0×10^4	1.0×10^4	1.0×10^4	7.2×10^4	50×10^4	3.6×10^4
4.0 ^e	0	0	0	0	0	0

^aAll readings taken with 20 psi at the nozzles.

^bM6 generator with one nozzle open.

^cM6 generator with three nozzles open.

^dM6 generator with six nozzles open.

^e4.0 μm and above no particles were detected.

Table 4

DOP AEROSOL CONCENTRATION PRODUCED
AS TESTED BY PHOTOMETER^a

Pressure (psi)	Relative Concentration (%) Produced by Generators					
	S2	M1M	M6-1 ^b	M6-2 ^c	M6-3 ^d	M6-6 ^e
5	1.3	0.28	1.6	2.8	3	7
10	6.8	0.7	4.7	8.0	13	30
15	12.0	1.0	7.1	14.0	25	46
20	15.0	1.3	11.0	20.0	31	54
25	19.0	1.5	12.1	22.0	38	62
30	21	1.52	12.0	26.0	40	70

^aRelative concentration based on Figure 9 and meter readings.^bM6 generator with one nozzle open.^cM6 generator with two nozzles open.^dM6 generator with three nozzles open.^eM6 generator with six nozzles open.

Table 5

DOP CONCENTRATIONS FOR SIX-NOZZLE GENERATOR^aWITH DILUTION SYSTEM SET ON 10^{-2}

Nozzles Open	Average 10-min Concentration ^b
1	8.6×10^7
2	17.0×10^7
3	24.5×10^7
4	29.9×10^7
5	33.3×10^7
6	34.0×10^7

WITH DILUTION SYSTEM SET ON 10^{-3}

Nozzles Open	Average 10-min Concentration
1	5.6×10^7
2	11×10^7
3	16.6×10^7
4	23×10^7
5	30×10^7
6	38×10^7

^aAll readings taken with 20 psi to the nozzles, $0.5 \mu\text{m}/\text{ft}^3$ of air/min and above using the particle counter.^bParticles/ ft^3 .

FIGURE 10 - Relative concentrations produced by the M1M and M6 (with one nozzle open) at different pressures.

FIGURE 11 - Relative concentration produced by the S2 and M6 (with two nozzles open) generators at different pressures.

FIGURE 12 - Relative concentration produced at different pressures by the M6 generator with one, two, three, and six nozzles open.

Table 5 shows that the M6 generator does produce more aerosol concentration as the nozzles are added. The 10^{-2} and the 10^{-3} ranges did not exactly agree (see Figure 13); however, the generator does produce at approximately the amount that would be expected. (It is believed that in the 10^{-2} range with five and six nozzles open, the particle counter was approaching its saturation point.)

CONCLUSIONS

The six-nozzle DOP generator performed reasonably well for the anticipated usage that it will receive at Mound Laboratory. We were pleased with the particle size distribution data which showed the majority of the dispensed DOP aerosol to be in the 0.3 to 0.8 μm range for the tests run.

The one-nozzle DOP generator did not perform as expected because the 1/8 in. nominal copper tubing from the inlet to the nozzle was too small. However, it can be used to supply the DOP aerosol concentration to our new automated HEPA Filter Scanner Tester (Figure 14).

This preliminary development effort indicates that a three-nozzle DOP generator with 1/4 to 3/8-in. i.d. tubing would be the optimum design for our use when clean air modules¹ are tested. Some thought also has been given to the use of a gross particle trap reservoir after the aerosol outlet and to changing the nozzle design by opening up the jet size.³

FIGURE 13 - Particle concentration produced by the M6 generator as determined by the particle counter at 10^{-2} and 10^{-3} dilutions.

ACKNOWLEDGMENTS

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FIGURE 14 - HEPA Filter Scanner Tester.

APPENDIX

Hot DOP generators are used to detect leaks in air filtration systems. Each generator requires 110 V ac and a source of compressed nitrogen. An aerosol of consistent particle size distribution is created by discharging a regulated quantity of DOP liquid into a heated area. The DOP is vaporized and reconstituted into a polydispersed aerosol by a small amount of nitrogen.

Cold DOP generators are used to detect leaks in air filtration systems. All that is required to operate is a source of clean compressed air. Based on the Laskin nozzle type apparatus, it creates a liquid aerosol concentration of consistent particle size distribution by shearing the DOP liquid with air.

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