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Performance of Multiple HEPA Filters Against Plutonium Aerosols

January 1 through June 30, 1974

by

John C. Elder
Harry J. Ettinger, Project Manager
Manuel Gonzales
Marvin Tillery

los alamos
scientific laboratory
of the University of California
LOS ALAMOS, NEW MEXICO 87544

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PERFORMANCE OF MULTIPLE HEPA FILTERS AGAINST PLUTONIUM AEROSOLS
JANUARY 1 THROUGH JUNE 30, 1974

by

John C. Elder; Harry J. Ettinger, Project Manager; Manuel Gonzales; and Marvin Tillery

ABSTRACT

The efficiency of a multiple HEPA filter system (three stages) has been evaluated using laboratory-produced plutonium oxide aerosols having activity median aerodynamic diameters (amad's) ranging from 0.22 to 0.44 μm. Extensive efforts have been made to improve the accuracy of assessing the efficiency of HEPA #3. These efforts have consisted of increasing the aerosol concentration challenging HEPA #3 and improving the assessment of the penetrating concentration. The more accurate measurements of HEPA #3 performance have resulted in the measurement of higher efficiencies for this stage. For the eight runs made during the reporting period, all HEPA #3 efficiencies were in excess of 99.99%. The average efficiencies measured for HEPA #1 and HEPA #2 were also in excess of 99.99%. The runs made during the reporting period indicated that the minimum-size aerosol that can be practically produced by centrifugal milling and ultrasonic agitation will have an amad $\sim$ 0.2 μm. The investigation of new techniques to produce plutonium aerosols having amad's $\leq$ 0.1 μm has been initiated.

The efficiency of filters that have been exposed to alpha irradiation for periods up to 12 months were measured. No decrease in performance could be detected in these filters.

I. SUMMARY

Performance of multiple stages of HEPA filtration for plutonium aerosols has been measured more accurately. The improvement in measuring techniques has led to a higher value for the measured protection factor of HEPA #3 filtration stage. This improvement in measurement was achieved by increasing the challenge aerosol concentration to HEPA #3 and by increasing the accuracy of measurement of penetrating aerosol concentration. The challenge concentration at HEPA #2 and #3 has been increased by using filters with lower efficiencies at HEPA #1 (but still satisfying the 99.97% minimum efficiency criterion), and by increasing the initial challenge aerosol concentration. Improvement in assessing the amount of aerosol penetrating HEPA #3 was made by increasing run time and by a more complete elimination of contamination. These efforts have indicated higher efficiencies for HEPA #3. Average efficiencies measured for each of the three stages of filtration have been in excess of

99.99% for these more recent runs. When these runs are averaged with earlier runs the average efficiency of HEPA #3 is 99.86%. With respect to particle size the performance of all stages has been in excess of 99.95% for all sizes of particles including the fraction of particles having aerodynamic diameters of less than 0.12 μm.

Initial measurements have been made of the efficiency of HEPA filters operated at one-half of rated flow. The efficiencies as a function of particle size have not been determined at this time. Overall efficiencies at one-half of rated flow are comparable to the overall efficiencies measured at rated flow.

Efficiency measurements were made on filters subjected to alpha irradiation for periods up to one year. These filters had up to 2.5 grams of $^{238}PuO_2$ collected as an aerosol. These efficiency measurements indicated no measurable deterioration in filter performance.

Results obtained in this study indicate that each stage of series-mounted, leak-free HEPA filters will perform above minimum requirements. These results also apply for aerosols having $D_{ae} \leq 0.22$ μm, (D_{ae}, Diameter of unit density sphere with the same gravitational settling velocity). Protection factors measured for these filters connected in series have consistently been in excess of 10^{13}.

Calibration of Andersen cascade impactors under conditions similar to operational conditions for this study has been started. In addition to determination of collection efficiency as a function of particle size for each stage, the effect of varying the collection surface will be measured.

II. EXPERIMENTAL DETERMINATION OF HEPA FILTER EFFICIENCIES AT RATED FLOW

Modifications have been made in experimental procedures to improve the measurement of third-stage efficiency. Modifications include the use of higher challenge concentrations, longer runs and lower efficiency filters at HEPA #1 to improve counting statistics. The most effective of these modifications is the use of filters with lower collection efficiency at HEPA #1. The filters used for the more recent tests had efficiencies measured by 0.3-μm DOP aerosols ranging from 99.970% to 99.978%, in contrast to efficiencies of at least 99.985% for previous tests. These lower efficiencies combined with the higher challenge concentrations and longer run times increased the activity concentration downstream of HEPA #3 by a factor of as much as 100 leading to a reduction in counting errors of about 75%. Counting accuracy has also been improved by reducing the contamination level by increased decay time (to eliminate contamination by radon and thoron daughters) and a more accurate assessment of background counts. A careful check of possible sources of contamination revealed an inherent alpha background in the fibrous glass filters used to sample downstream of HEPA #3. Background counts for these filters are about twice as high as the backgrounds measured with Type AA Millipore filters. The difference in background will be accurately assessed in order to correct earlier reported HEPA #3 efficiencies. This effect will result in higher efficiencies for HEPA #3 as the counts used to evaluate the penetrating aerosol are of the same order of magnitude as the background.

To present a severe challenge to the HEPA filters, extensive efforts were made to generate smaller aerosols using current generation techniques. These efforts have involved milling $^{238}PuO_2$ for up to 210 hrs in a centrifugal mill and ultrasonic agitation of the suspension for as long as 48 hrs. These efforts have produced only slightly smaller aerosols and seem to represent the practical limit of this technique. The smallest aerosol generated with the technique had an activity median aerodynamic diameter (amad) of 0.22 μm. These results indicate that different generation techniques will be required to generate aerosols having amad's ≤ 0.1 μm.

TABLE I

EFFICIENCIES OF HEPA FILTERS

Run	HEPA Filter Stage	Plutonium Aerosol amad (μm)	σ_g	Activity Concentrations d/s-m^3	HEPA Filter Efficiency (%)
P4-10	0	0.44	2.1	1.07×10^{10}	
	1	0.53	2.3	1.66×10^{6}	99.99+
	2	0.42	1.7	5.83×10^{1}	99.99+
	3	-		4.23×10^{-3}	99.99+
P4-11	0	0.43	3.4	1.94×10^{10}	
	1	0.47	2.4	1.94×10^{6}	99.99
	2	0.39	1.9	6.49×10^{1}	99.99+
	3	-		3.87×10^{-3}	99.99+
P4-12	0	0.33	3.6	1.38×10^{10}	
	1	-		1.09×10^{5}	99.99+
	2	0.15	1.9	1.07×10^{1}	99.99
	3	-		9.67×10^{-4}	99.99
P4-13	0	0.27	3.9	1.04×10^{10}	
	1	-		9.29×10^{4}	99.99+
	2	0.20	2.5	9.15×10^{0}	99.99
	3	-		6.11×10^{-4}	99.99+
P4-14	0	0.22	2.6	1.68×10^{10}	
	1	-		2.54×10^{5}	99.99+
	2	0.28	2.3	1.41×10^{1}	99.99+
	3	-		1.10×10^{-3}	99.99+
P4-15	0	0.26	3.2	1.12×10^{10}	
	1	0.29	2.2	9.12×10^{4}	99.99+
	2	-		1.29×10^{1}	99.98
	3	-		1.14×10^{-3}	99.99
P4-18	0	0.37	3.2	9.29×10^{9}	
	1	0.30	2.5	4.23×10^{4}	99.99+
	2	0.22	2.5	2.90×10^{0}	99.99+
	3	-		1.69×10^{-4}	99.99+
P4-19	0	0.32	3.7	5.40×10^{9}	
	1	0.30	2.1	1.08×10^{5}	99.99
	2	0.28	2.4	1.31×10^{1}	99.98
	3	-		3.25×10^{-4}	99.99

Utilizing these modifications and challenge aerosols having amads as small as 0.22 μm, eight experimental runs have been made. Results of these runs are shown in Table I. Average efficiencies measured for the first and second HEPA filter stages were in excess of 99.99% with a minimum efficiency of 99.98%. For the third stage of filtration (HEPA #3) the efficiencies were consistently higher than for earlier runs. For HEPA #3 the minimum efficiency was in excess of 99.99%. These measured efficiencies are well above the 99.8% normally assumed for second and third stage HEPA filters. A summary of all efficiencies measured for small particles to this time is given in Table II. All results shown in this table are for operation at the design flowrate of 1.18 x 10^{-2} m^3/sec (25 cfm). Inclusion of the eight recent runs increases the average efficiency of HEPA #3 to 99.86%. The high alpha count backgrounds noted for the media used to sample downstream of HEPA #3 have not been considered in determining these efficiencies. This correction will be applied when the average value of the background is determined, and will increase the efficiencies of HEPA #3.

With respect to particle size the highest penetration was observed for particles having aerodynamic diameter $\leq$ 0.12 μm. (Table III) This size material is represented by the particles collected on stage seven and the backup filter of the Andersen cascade impactor. For the eight recent runs the collecting efficiency measured for particles in this size range was always better than the minimum performance criteria of 99.8%.

TABLE II

SUMMARY OF HEPA FILTER EFFICIENCIES
(17 tests to date)

Stage	amad Range (μm)	HEPA Filter Efficiency (%) Minimum	Maximum	Avg.
1	0.22 - 0.66	99.99+	99.99+	99.99+
2	0.29 - 0.53	99.98+	99.99+	99.99+
3	0.15 - 0.42	99.50	99.99+	99.86

TABLE III

AVERAGE EFFICIENCY WITH RESPECT TO PARTICLE SIZE FOR RUNS P4-10 THROUGH P4-19 (TOTAL OF 8 RUNS)

Impactor Stage	Size Interval D_{ae} (μm)	Mean Efficiency HEPA #1	HEPA #2
0	>5.40	99.999	99.972
1	3.40-5.40	99.999	99.995
2	2.30-3.40	99.999	99.997
3	1.54-2.30	99.999	99.999
4	.96-1.54	99.994	99.998
5	.44- .96	99.993	99.988
6	.22- .44	99.995	99.989
7	.12- .22	99.998	99.963
MF2	<.12	99.998	99.958

A more accurate assessment of the filter's performance against small particles requires aerosols having a large amount of activity associated with small particles. Initial attempts to provide an aerosol having an amad $\leq$ 0.1 μm with prolonged grinding has met with limited success. A second method being investigated is the drying of a plutonium nitrate solution to produce small particles. These particles are then resuspended in water to produce a hydrosol of very small particles. This method has been reported to produce particles having physical sizes smaller than 0.01 μm.

III. DETERMINATION OF HEPA FILTERS EFFICIENCY AT ONE-HALF RATED FLOW

The predominant mechanisms of collection in filtration are inertial impaction and diffusion.[1] Inertial impaction is important for larger particles and efficiency of collection by this mechanism is directly proportional to the velocity through the filter. Diffusion collection is important for small particles, and efficiency of collection is inversely proportional to the

TABLE IV

EFFICIENCIES OF HEPA FILTERS AFTER EXTENDED EXPOSURE TO $^{238}PuO_2$

Filter Number	Total Loading (g-$^{238}PuO_2$)	Exposure Time (days)	Test #	Filter Efficiency: Before Exposure amad (μm)	σ_g	Efficiency (%)	After Exposure amad (μm)	σ_g	Efficiency (%)
415	0.50	379	1	0.7	2.3	99.99+	0.8	9.3	99.99+
			2	1.3	2.9	99.99+	0.7	5.2	99.99+
			3	1.3	2.7	99.99+	0.7	6.4	99.99+
414	0.73	385	1	0.6	2.2	99.99+	0.8	5.0	99.99+
			2	0.8	2.7	99.99+			
			3	1.6	2.7	99.99+			
430	2.50	372	1	0.7	2.1	99.99+	0.6	4.0	99.99+
			2	0.8	2.2	99.99+			

velocity through the filter. The result of these two velocity effects is a particle size of maximum penetration for a given set of operational conditions. For a different flowrate through the filter the particle size of maximum penetration will vary. By varying the filtration area of successive stages of filtration it should be possible to increase the overall collection efficiency of a series filter installation. It has been shown in this study that a slight decrease in efficiency occurs for the smallest size fraction of the plutonium aerosol at normal flow. Efficiency measurements are currently being conducted at one-half rated flowrate to evaluate the effect of lower flowrate. Preliminary data based on two runs show overall efficiencies comparable to the rated flowrate efficiencies. Data on efficiency as a function of particle size has not been completely analyzed at this time. The half flow runs should be completed during the next reporting period. Results of these runs will be used to determine operational conditions for future experiments.

IV. RETEST OF HEPA FILTERS AFTER EXTENDED EXPOSURE TO $^{238}PuO_2$

A theoretical study reported in an earlier report[2] indicated that the alpha dose ($\sim 3 \times 10^{20} \alpha/m^2$) from a $^{238}PuO_2$ particle on a glass fiber for one year would be in the range of doses ($\sim 10^{20} \alpha/m^2$) that caused fine cracks in Pyrex glass. As these results indicated possible degradation of filter performance after prolonged exposure to alpha radiation, an experimental investigation was carried out using the first-stage filters that had been exposed to ^{238}Pu alpha particles for periods of approximately one year. Three filters were retested. The $^{238}PuO_2$ loadings on these filters were 0.50, 0.73, and 2.50 g. The pressure drop across the filter loaded with 2.5 g was 229.5 Pa (2.34 cm of H_2O). In normal field operations a filter would receive a higher mass loading and differential pressure increase before it was changed. However, much of this loading would be from inert material in the air stream, and the test filters probably received alpha doses comparable to those encountered in the field. The results of the retest of these filters are given in Table IV. Total loading of filters 414 and 415 was the result of three separate tests during a short time span. The 2.50 mg loading of filter 430 was accumulated in two tests. Results of these tests are listed in the 'before exposure' column. After a storage period for radiation exposure, filter 415 was retested against three test

aerosols. The other two filters were tested against one aerosol. There is no detectable difference in the overall performance of these filters after irradiation. In all cases the filters performed with efficiencies in excess of 99.99%.

This test may not have been quite as severe as field exposure, as these filters were stored with no flow through the filters during the year. In field operations the long-term flexure of the fibers by the flow stream and the higher pressure differential across the filter might lead to more fiber breakage. A decrease in the length of the fibers should not affect filtration if the mechanical integrity of the filter is maintained. We conclude that alpha irradiation of the glass fibers and adhesive, in the range of α dosage likely to be encountered in normal operations, will result in no significant deterioration in filter performance.

Since there was no decrease in filtration efficiency, plans to dismantle the filters for close inspection were dropped. Visual inspection of the filter faces showed distinct discoloration by PuO_2 in all three filters.

V. FIELD SAMPLING OF PLUTONIUM AEROSOLS

Alpha spectrometry of cascade impactor samples taken in the process ventilation ducts of DP West facility LASL was carried out during this report period. The purpose of this study is to determine any significant differences in the size distributions of the particles containing ^{238}Pu and ^{239}Pu. Analysis was carried out with silicon surface barrier detectors as described in an earlier progress report.[2] The normal sampling period for a single impactor run spans several work days. Thus, the particle size distribution represents the average for process procedures underway during the sampling period. Table V gives activity ratios for size ranges separated by the cascade impactor. There is no consistent ratio with respect to particle size except for the smallest size range (<0.43 μm) where the ^{239}Pu predominates. The size distributions derived from these data are given in Table VI. The analysis of cascade impactor data is based on the assumption that the size distribution is log-normal. These data indicate that size distribution given by the total activity is a reasonable estimate of the size distribution for each individual isotope. However, the wide variation observed in isotopic ratios would make it impossible to use average values for the isotopic ratio of the

TABLE V

ISOTOPIC RATIOS: ACTIVITY OF $^{238}Pu/^{239}Pu$

Cascade Impactor Stage	Size Interval D_{ae} (μm)	Ratios Run Number One	Run Number Two	Run Number Three
0	>11.0	0.48	1.50	1.00
1	7.0-11.0	0.64	0.91	1.00
2	4.7- 7.0	0.57	0.32	1.10
3	3.3- 4.7	0.58	0.64	3.30
4	2.1- 3.3	0.70	2.00	6.10
5	1.1- 2.1	0.68	2.20	4.50
6	0.65-1.1	0.58	1.20	5.90
7	0.43-0.65	0.26	0.52	2.40
Filter	<0.43	0.13	0.33	0.37

TABLE VI

SIZE DISTRIBUTIONS WITH RESPECT TO TOTAL ACTIVITY AND INDIVIDUAL ISOTOPES

Run Number	^{238}Pu amad (μm)	^{238}Pu σ_g	^{239}Pu amad (μm)	^{239}Pu σ_g	Total Activity amad (μm)	Total Activity σ_g
One	3.1	1.4	3.1	1.4	3.1	1.4
Two	2.6	1.3	3.1	1.4	2.8	1.4
Three	1.8	1.5	1.8	1.8	1.8	1.6

aerosol challenging the air cleaning systems of this facility.

VI. COLLECTION SURFACE EFFECTS ON REBOUND IN CASCADE IMPACTORS

The rebound study discussed in the previous progress report[2] is being continued on a low-priority basis. During this quarter additional measurements have been made. However, sampling difficulties were encountered with all runs. These difficulties seem to be associated with the plugging of jet holes by collection of fibers from the exhaust air ventilation ducts. A difficult aspect of this study has been the selection of a reference collection surface for comparison with other surfaces. For the initial studies a glass fiber filter was used for a reference surface. It has recently been observed[3] that fibrous mat filters modify the collection efficiency as the airstream penetrates into the filter so many particles are removed by filtration. An impactor calibration study (discussed in Section VII) is being initiated to resolve this problem.

VII. CALIBRATION OF ANDERSEN CASCADE IMPACTOR

The Andersen cascade impactor is used to size the plutonium aerosols in both experimental and field portions of this study. For some of these tests the impactor is operated at three times the design flowrate to gain more information on small particles. The calibration used for the high flowrate is based on one study[4] in which stages 2-6 were calibrated. For the other stages the effective cutoff diameter (ECD, diameter of unit density spherical particle for which collection efficiency is 50%) is calculated. Because of recent comments on the performance of the instrument by other workers, we have decided to calibrate the instrument under our operational conditions.[5,6,7] This will include calibration at flowrates of one and three cubic feet per minute (0.000 47 m^3/sec and 0.0014 m^3/sec). For a flowrate of 1.0 cfm the ECD provided by the manufacturer has been used. This calibration has been substantially reconfirmed by several independent workers.[8,9] In this laboratory the calibration has been spot checked using monodisperse polystyrene latex (PSL) particles.[10] These checks showed reasonable agreement with the manufacturers values. However, to fully resolve this question, a detailed calibration will be carried out at both flowrates utilizing several collection surfaces. The effect of impactor collection surface will be carefully considered as previous calibrations have not been carried out with the surfaces used in the LASL plutonium filtration study. For most of our samples membrane filters have been utilized as most types can be dissolved to permit dilution of high-level samples to reasonable counting levels.[11] The effect of electrostatic attraction, reportedly a major collection mechanism in membrane filters, will not be investigated as condensation aerosols having low charge will be used for the calibration. This should be representative of the experimental aerosols as the dense radiation fields associated with the plutonium aerosols should effectively discharge the particles.

Features of various calibration methods that have been used in the past are listed in Table VII. For the initial part of the calibration study the method described by Swartz was used.[12] This method was chosen because it was believed that a polydisperse wax aerosol would be easy to generate and characterize. Initial attempts to use this aerosol for calibration have not been satisfactory as the lower stages overloaded before enough particles could be collected on the upper stages. Step-wise rotation of the lower stages failed to alleviate this problem. Sizing of the smaller particles collected on the lower stages has also proven to be quite difficult. Solubility of wax particles in the clearing solution used for membrane filters disqualify this aerosol and analysis method for collection on this surface. Initial work is currently underway

TABLE VII

SUMMARY OF SELECTED CASCADE IMPACTOR CALIBRATION METHODS

Investigator	Impactor	Aerosol	Collection Stage Coating	Sizing Method: Source Aerosol	Sizing Method: Collection Stage	Comments
Flesch[9]	Andersen 6 stage @ 1 cfm	Monodisperse methylene-blue, PSL*	none	L.M.,E.M.*, impinger	Mass Analysis by colorimetry or nephelometer	Stages 0, 1, 6, 7 not calibrated
Swartz[12]	Single jet 6 stage	Polydisperse red paraffin	none	N.A.*	L.M. to 0.5 μm	Limited to d >0.5 μm
Couchman[13]	Cohen 4 stage,2 identical stages	Polydisperse copper spheres	Vaseline	L.M.	L.M., Centrifuge	Limited to d >0.5 μm; ΔP across stage not same for calibration sampling
May[8]	Modified 6 stage Andersen	Polydisperse fluorescent bacteria	Agar and glass	N.A.*	L.M.	Stage 1 and 2 modified limited to d >0.5 μm
Hu[4]	Andersen 6 stage @ 3 cfm	Monodisperse PSL	Gelman Type A filter	N.A.*	Royco	Stages 0, 1, 7 not calibrated
Ranz and Wong[14]	Single stage, variable velocity	Monodisperse glycerol	none	Owl-G-2	Gravimetric	Varying velocity
Andersen[15]	Andersen 6 stage @ 1 cfm	Polydisperse carnauba wax	none	L.M.	L.M.	

* L.M. = light microscope
E.M. = electron microscope
PSL = polystyrene latex particles
N.A. = not applicable

to construct a La Mer type monodisperse aerosol generator[16] for paraffin aerosols. This generator should produce sufficient aerosol in each size interval to permit gravimetric analysis of collection efficiency.

VIII. FUTURE WORK

1. The development of generator techniques to provide challenge plutonium aerosols with amad's $\leq$ 0.1 μm will continue.

2. Sampling techniques for size characterization of these small aerosols, and determination of filter performance when challenged by these aerosols will continue.

3. Tests at flowrates below rated flow will be carried out to determine the feasibility of varying the area of successive

stages to increase overall collection efficiency.

4. Field sampling with dual impactors will continue at the LASL DP West facility on a time-available basis. These samples will be analyzed for isotopic ratios, particle size distribution of challenge aerosol, and particle rebound in the Andersen impactor.

5. The calibration of the Andersen cascade impactor will continue.

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