CONFINEMENT OF AIRBORNE ACTIVITY FROM MELTED ANTIMONY SLUGS*

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Abstract

During charge-discharge operations of a Savannah River reactor on November 9, 1970 antimony in an antimony-beryllium source rod melted while the rod was suspended in air. The failure released some 85,000 curies of activity, about 6250 curies of which was carried by ventilating air to the reactor building's confinement filters. The filters functioned extremely well, retaining more than 99.999% of the activity. Only 3 millicuries escaped to the environs – representing 0.63% of the maximum permissible concentration at the plant boundary.

The ventilation exhaust system for the reactor building includes five confinement filter compartments to contain radioactivity in the event of a reactor accident. Exhaust fans maintain a negative pressure in the reactor room, the ducts to the filters, and the compartments containing the filters. Each compartment contains moisture separators, particulate filters, and halogen adsorbers. It is unshielded, has dimensions of 9 × 22 × 25 ft., weighs ten tons, and is designed for remote removal from the reactor building roof 55 feet above ground level.

Four compartments were on-line when the accident occurred. During the subsequent three-month building cleanup period the air flow gradually decreased as the filters accumulated about three times their normal dust loading. No activity escaped from the filters during the cleanup period. Immediately after the accident the maximum radiation level at the side of the hottest filter compartment was 70 R/hr, which was reduced through activity decay to 25 R/hr by the time of replacement. Removal of the contaminated compartments began on February 9, 1971 following completion of the reactor room cleanup. Compartments were pulled to the edge of the roof, lifted from the roof with a crane, and placed on a special railroad flat car for movement to an isolated location. Replacement compartments were installed, new filters tested, and the system returned to normal on February 26, 1971.

Initial entry into the contaminated compartments to recover samples for detailed analyses was accomplished on March 20, 1972. Activity distributions among particulate filters were measured for the most heavily and the most lightly contaminated compartments. One compartment had a variation of ±4% about its average, while the other had a +18% to -30% variation. Radiation damage was minimal in the most heavily contaminated compartment which had an exposure of about 10^6 R.

Introduction

On November 9, 1970 an irradiated antimony-beryllium source rod overheated while it was suspended in air above K reactor at the Savannah River Plant. The reactor had been shut down the previous day and routine charge-discharge operations were in progress. Failure occurred with the rod suspended by the charge machine while the rod's housing was being replaced in the reactor. At the

* Information in this document was developed during the course of work under Contract AT(07-2)-1 with the U. S. Atomic Energy Commission.

time of failure, the source rod contained about 119,000 Ci of activity, largely ^{122}Sb and ^{124}Sb (table I). The overheating caused part of the antimony slug and aluminum cladding to melt, releasing about 85,000 Ci in the reactor room.* Approximately 90% of the released activity remained dispersed on the charge machine itself, 2% was distributed on the reactor plenum top and reactor room floor and walls, and 8% was carried by ventilating air to the reactor building's confinement filters.

Decontamination of the charge machine and reactor room, and replacement of the contaminated filter compartments required about 3½ months. The reactor was returned to normal operation on February 26, 1971.

Discussion

Description of the Confinement System

Confinement facilities for the Savannah River reactors were installed in 1962[2]. Airborne particulate and halogen contamination is collected by the confinement system (figure 1). Because the building is not gastight, the ventilation system is designed so that three exhaust fans with redundant power supplies continuously maintain a negative pressure in the process areas, the ducts to the filters, and the compartments containing the filters. Air exhausted from the reactor room, the below-grade process area, and the purification area is channeled into a common plenum before passing through the filter compartments. Because complete mixing does not occur in the plenum, unequal activity distribution occurs among the different compartments depending on the building area in which activity is released. The filters are on line continuously to avoid the necessity of opening and closing dampers after an accident takes place.

The filters are contained in five aluminum compartments that operate in parallel and are located on the reactor building roof 55 feet above ground level. Each compartment weighs 10 tons and is 9 feet wide, 22 feet long, and 25 feet high. Each contains three banks of filters in series (figure 2):

1) Moisture Separators consisting of two-inch-thick mats woven from stainless steel wire wrapped with "Teflon" fibers. Twenty units 2 feet square are used in each compartment.
2) Particulate Filters designed to remove more than 99% of all particles 0.3 micron or larger. Each filter is made of an 18-mil-thick glass-asbestos felt sheet folded over corrugated aluminum inserts into closely spaced pleats forming a unit 2 feet square by 11½ inches thick. Thirty-two units are used in each compartment.
3) Halogen Adsorbers consisting of one-inch-thick beds of 10 to 14-mesh activated coconut-shell charcoal enclosed between perforated metal sheets that are pleated to form air pockets. The beds are stacked together to form a unit 2 feet square by 11½ inches thick. Thirty-two units are used in each compartment.

Each filter compartment sits on a rail dolly that can be positioned from ground level by a system of cables and pulleys. Both the inlet and outlet nozzles of a compartment face the building so that they can be attached to the ventilation system by fitting over nozzle extensions on the building. Inflatable seals at the nozzles eliminate leakage. A flapper door at each compartment nozzle seals the compartment when it is disconnected from the building ventilation system. When a compartment is delatched from the building a catch on each flapper door releases and allows the door to swing down and to be held shut by two refrigerator-type latches.

* Activity data in this report have been revised from estimates cited earlier[1] which were based on the results of preliminary radiation measurements.

Since no shielding is provided for the compartments, remote removal facilities were incorporated in their design. Latching and sealing controls and positioning cables are located at ground level, shielded by the reactor building from possible radiation from the filter compartments. If a compartment becomes contaminated, it can be disconnected remotely from the ventilation system, pulled to the edge of the 55-foot high roof, picked up by a crane, placed on a railroad car, and removed from the area.

Confinement System Performance

About 6250 curies was released in particulate form to the confinement filters, but only 3 millicuries of this activity escaped from the building exhaust stack. The confinement filters retained more than 99.999% of the activity. Levels of radioactivity in the atmosphere at the stack and at the plant boundary resulting from stack releases of ^{124}Sb were estimated by the Savannah River Laboratory. The maximum permissible concentration (MPC) for ^{124}Sb is 5×10^{-8} $\mu Ci/cc$ [3]. A 3-millicurie release represents 2.1 MPC at the stack and 6.3×10^{-6} MPC at the plant boundary. If the confinement filters had not been present 6250 curies would have been released, representing 4.4×10^{6} MPC at the stack and 13.2 MPC at the plant boundary.

The initial maximum radiation level observed at the side of the hottest filter compartment was about 70 R/hr. This had decreased to 25 R/hr when the filter compartments were removed from the building about three months after the accident. Compartment K-4 was off-line for routine maintenance, but the other four filter compartments were on-line at the time of the accident, and the total flow of exhaust air through them was 86,400 cfm. The four contaminated compartments remained in service during the three months following the accident; the flow gradually decreased to 61,000 cfm. Table II shows individual filter data prior to the accident and at the time the compartments were removed.

The major filter pluggage was experienced in the moisture separators; this was similar to normal moisture separator pluggage that has been experienced at SRP after similar service lifetimes and is not attributed to the accident. Pluggage due to an accumulation of an oily substance and particulate matter on the upstream face normally limits moisture separator service to about 9 months. Plugged moisture separators normally are removed from the compartments and cleaned with steam jets to prepare them for continued service[4].

Estimates made by Savannah River Laboratory personnel, based on radiation measurements taken after the filter compartments were removed from the building, indicate that the activity was not equally distributed among filter compartments (table III). The two compartments nearest the reactor room exhaust duct contained 95% of the total activity released to the ventilation system. Activity measurements in filter compartment K-3 indicate that the moisture separators retained about 15% of the activity while the particulate filters retained the remaining 85%.

Filter Compartment Replacement

Replacement of the contaminated filters was desirable because their continued use would result in radiation exposure for personnel working at ground level outside the reactor building and even higher exposure for personnel replacing plugged filters.

Spare filter compartments are not maintained at SRP, but replacements, which had been in service previously were obtained from an out-of-service reactor building. These compartments were removed from the roof, refurbished, pressure tested, equipped with new filters, and trucked to K Area. The height of the load required clearance of some overhead obstructions. These overhead obstructions were cleared by lifting the load over them, by removing or burying the obstructions, and by cutting new roads below grade level (figure 3).

Removal of the contaminated compartments from K Area began on February 9, after completion of the reactor room cleanup, and was completed by February 12, 1971. Individual compartments were moved remotely to the edge of the roof. An expanding urethane foam was sprayed into each of the compartment nozzles to fix any loose contamination and provide a seal if a compartment flapper door failed to seal (figure 4). This was done with several ten-cubic-foot "Insta-Foam FROTH PAKS" (Trademark of Insta-Foam Products, Inc.). Applications of the foam on the 56-inch diameter nozzles and flappers required approximately 1½ minutes in a maximum radiation field of 5 R/hr.

The compartments were removed from the roof by a 75-ton motor crane equipped with 120 feet of boom (figure 5). A special remote-handling lifting hook with long tag lines was employed. The most critical crane operation was to lift the compartment straight up off the movable rail dolly. The crane operator, located at the base of the building, could not see the load. Two spotters with binoculars and short wave radios directed the crane operations; one spotter was located on an adjacent building roof and the second spotter was supported in a basket by a second crane. Once the compartment was clear of the rail dolly, the compartment was lowered to a modified railroad car (figure 6). The maximum radiation exposure rate for the crane operator was 200 mR/hr.

The railroad car support frame sustained the compartment so that no tie-downs were required. The train was made up of the modified car, three spacer cars and a locomotive (figure 7) for movement to the storage area. The train was preceded by a track motor car with personnel to visually inspect the track and line switches, to spike facing switch points, and to open security fence gates. Primary plant road crossings on the route were blocked by Patrol personnel to eliminate the possibility of radiation exposure to personnel at those locations. A second track motor car followed the train to restore switches to their normal operating positions and to carry a Health Physicist who surveyed the track to ensure that no contamination was released. Movement of each load required one hour. The maximum radiation exposure rate to the railroad crew was 30 mR/hr.

The contaminated compartments were removed from the railroad train with a 100-ton crawler crane equipped with 65 feet of boom and the special remote-handling lifting hook with tag lines (figure 8). Maximum radiation exposure rate for the operator of this crane was 400 mR/hr. The four contaminated compartments are now stored behind an out-of-serivce reactor building where they will remain until reclamation operations are desired (figure 9).

Installation of the refurbished compartments was completed on February 12 and 14. After leak testing the particulate filters and carbon filters, the compartments were connected to the building ventilation system on February 18 and 26, thereby returning the K reactor confinement system to normal.

Inspection of Filter Compartments

Contaminated compartments K-6 and K-3 were entered on March 20, 1972 to obtain sample materials for detailed examination of the effects of irradiation on filter components and to determine the distribution of activity within the filter compartments (figure 10). Results of laboratory studies of the samples are included in another conference paper [5].

In each compartment, a radiation profile of the particulate filters was obtained to determine the activity distribution among the filters. A specially constructed probe assembly consisting of a 1-inch diameter × 3/4-inch-long NaI(Tl) crystal attached to a 1-inch-diameter photomultiplier was collimated with a lead shield (figure 11). The shielded probe, operated from the floor of the filter compartment by ropes and pulleys, was positioned at the center of each particulate filter while a count was obtained. Instrumentation used with the probe consisted of a preamplifier, amplifier, single channel analyzer, count rate meter, preset timer, scaler, and high voltage supply.

Selected filter units were removed from the compartments to permit sampling of filter media and to obtain radiation readings from individual filter units to confirm values obtained from the radiation profile measurements. Each filter unit removed from the compartments was placed in a constant geometry fixture (figure 12) for independent measurement of the radiation level with an ionization chamber contained in a portable, low-range, gamma survey instrument.

Compartment K-6 originally retained approximately 20 curies after the source rod failure. By March 20, 1972 the working radiation rate inside this compartment was approximately 10 mR/hr with smearable contamination of approximately 300 cpm β-γ. The shielded probe was positioned in front of each filter and a 10-second count obtained. A variation of +18% to -30% from the average was measured. The filters on the left side (looking from downstream) had less activity than those in the center or on the right side (figure 13). The range of +18% to -30% measured inside the compartment with the shielded probe was in good agreement with the range of +23% to -26% determined from individual radiation measurements taken on 11 filters removed from the compartment and tested in the constant geometry fixture. Although relative concentrations measured by the two methods differed by as much as 16%, as shown in figure 13, the good agreement between the ranges verifies the validity of the in-compartment measurements with the shielded probe. This verification was important because removal of several individual filters from compartment K-3 for testing in the constant geometry fixture would have required appreciable personnel exposure.

Compartment K-3 originally contained approximately 3060 curies from the failed source rod. Working radiation rates and smearable contamination inside this compartment were proportionately greater than those in compartment K-6 (table IV). Activities measured by the shielded probe inside the compartment during a 1-second count of each filter are shown in figure 14. The activity distribution was much more uniform (a variation of only ±4%) than that in compartment K-6. Personnel exposures were minimized by using the in-compartment measurements to determine activity distribution; only the filters removed for sampling (one of each type) were surveyed in the constant geometry fixture.

Visual inspection of the filters indicated that no radiation damage had occurred to the "Teflon" fluorocarbon resin (Du Pont trademark) in the moisture separators or to the particulate filter media. An integrated dose of 6×10^6 R for the particulate filters and 7×10^5 R for the moisture separators was calculated when samples were obtained from the K-3 compartment. The foam nozzle seals were also observed to be in good condition.

Based on radiation levels and working conditions encountered in this work, reconditioning of compartments K-5 and K-6 would be possible, but reclamation of compartments K-3 and K-4 would be impractical. No compartments will be reclaimed at this time because they are not now required for plant operations.

References

1. J. W. Joseph, Jr. and J. W. Little, Jr., "Activity Confinement and Decontamination After Failure of an Antimony-Beryllium Source Rod," ANS Transactions, Supplement No. 2 to Vol 14, pp 43-44. Conference on Reactor Operating Experience, Denver, Colorado, August 1971.
2. J. A. List, "The SRP Production Reactor Containment Program," Eighth AEC Air Cleaning Conference, TID-7677, pp 273-279 (October 1963).
3. "Maximum Permissible Body Burdens and Maximum Permissible Concentrations of Radionuclides in Air and in Water for Occupational Exposure," U. S. Department of Commerce, National Bureau of Standards, Handbook 69 (June 5, 1959).
4. W. S. Durant, "Performance of Airborne Activity Confinement Systems in Savannah River Plant Reactor Buildings," Ninth AEC Air Cleaning Conference, CONF-660904, pp 348-367 (January 1967).
5. L. R. Jones, "Effects of Radiation on Reactor Confinement System Materials," presented at Twelfth AEC Air Cleaning Conference (1972).

TABLE I. SOURCE ROD INVENTORY AT TIME OF FAILURE

Isotope	Half-Life	Curies
Sb-122	2.8 days	70,000
Sb-124	60.2 days	37,100
Sb-125	2.7 years	500
Te-123m	117 days	3,300
Te-125m	58 days	8,000
		118,900

TABLE II. CONFINEMENT FILTER BEHAVIOR

Compartment	Flow, cfm		Individual Filter ΔP, in. H_2O*			
			Moisture Separator		Particulate Filter	
	Prior	At Removal	Prior	At Removal	Prior	At Removal
K-2	20,600	15,200	1.22	4.39	0.66	1.04
K-3	28,400	18,700	1.04	3.40	0.62	0.84
K-5	17,000	10,900	2.28	6.36	1.26	1.98
K-6	20,400	16,200	2.0	3.91	1.14	1.51

* Normalized to 20,000 cfm.

TABLE III. ACTIVITY DISTRIBUTION, CONFINEMENT FILTER COMPARTMENTS

Compartment	Initial Activity		Distribution of Retained Activity
	Curies	% of Total	
K-2	2873	46	
K-3	3061	49	{ Moisture Separators, 15% Particulate Filters, 85%
K-5	294	4.7	
K-6	19	0.3	
Total →	6247		

TABLE IV. COMPARTMENT K-3 RADIATION RATES

Location in Compartment	General Area Radiation, mR/hr	Smearable Contamination, CPM β-γ
Between moisture separators and particulate filters	800 (2,000 - 3" from moisture separators; 1,500 - 3" from particulate filters)	20,000
Between particulate filters and halogen adsorbers	700	2,000
Between halogen adsorbers and effluent nozzles	80	2,000

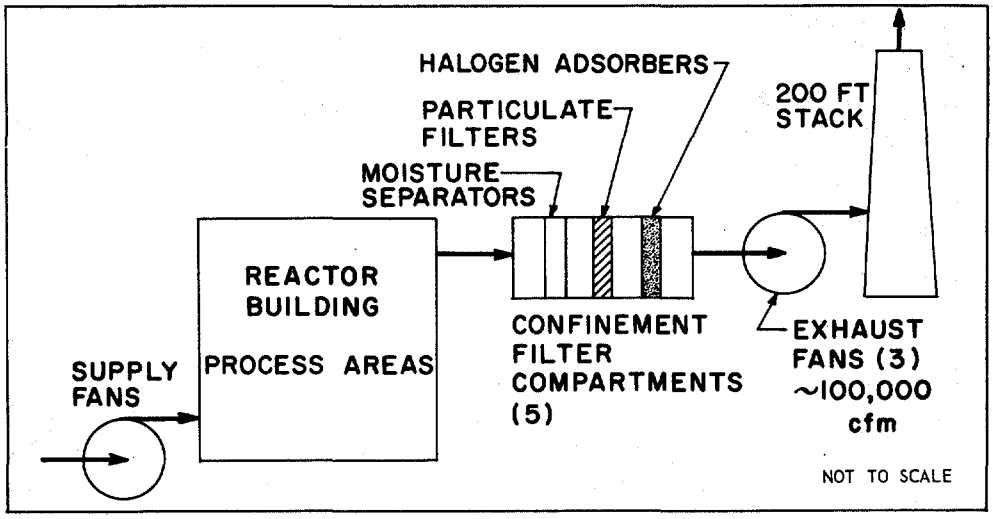

FIGURE 1. VENTILATION SYSTEM

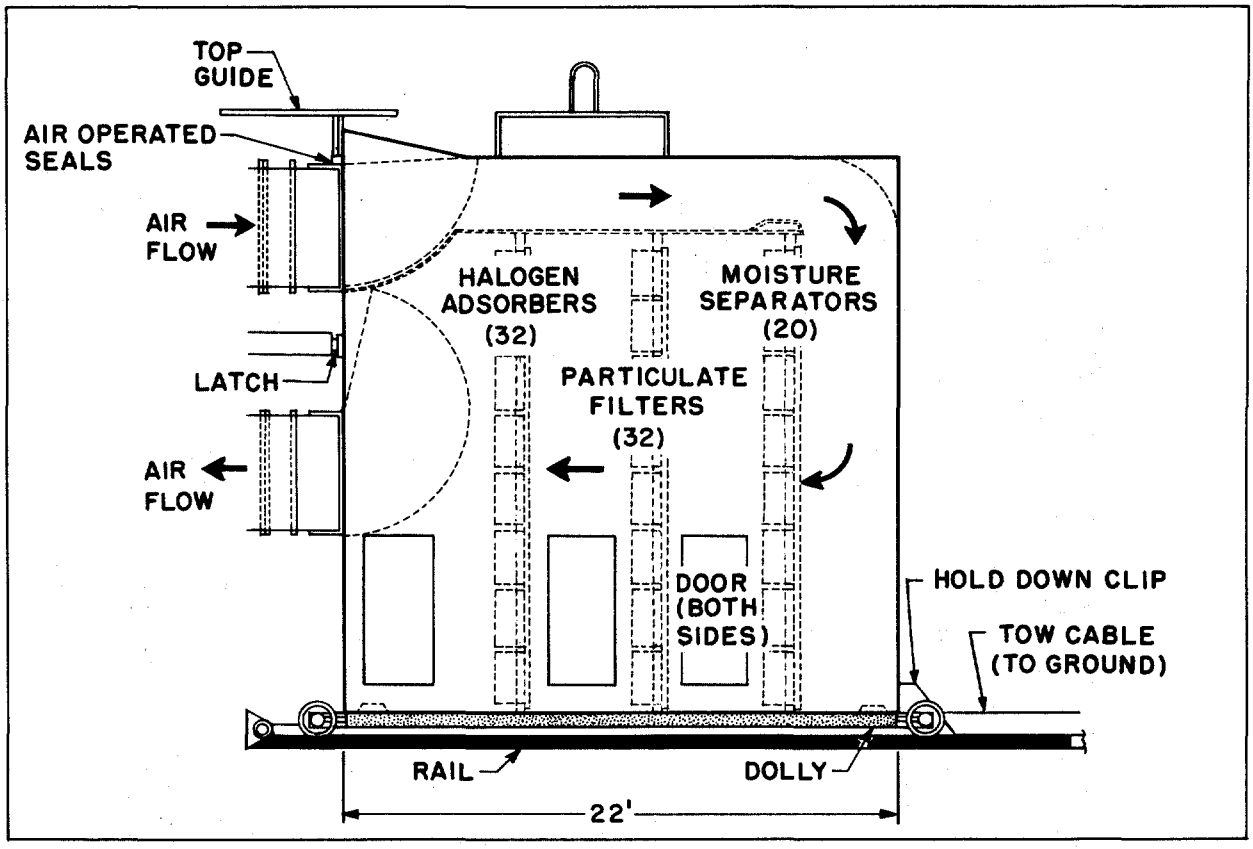

FIGURE 2. CONFINEMENT FILTER COMPARTMENT

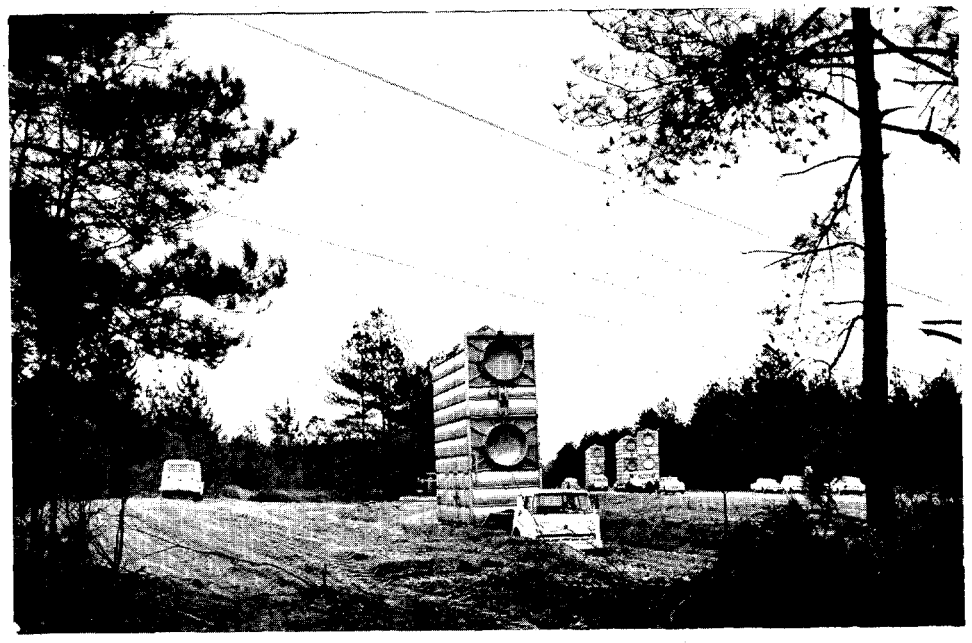

FIGURE 3. REPLACEMENT FILTER COMPARTMENTS EN ROUTE TO K AREA

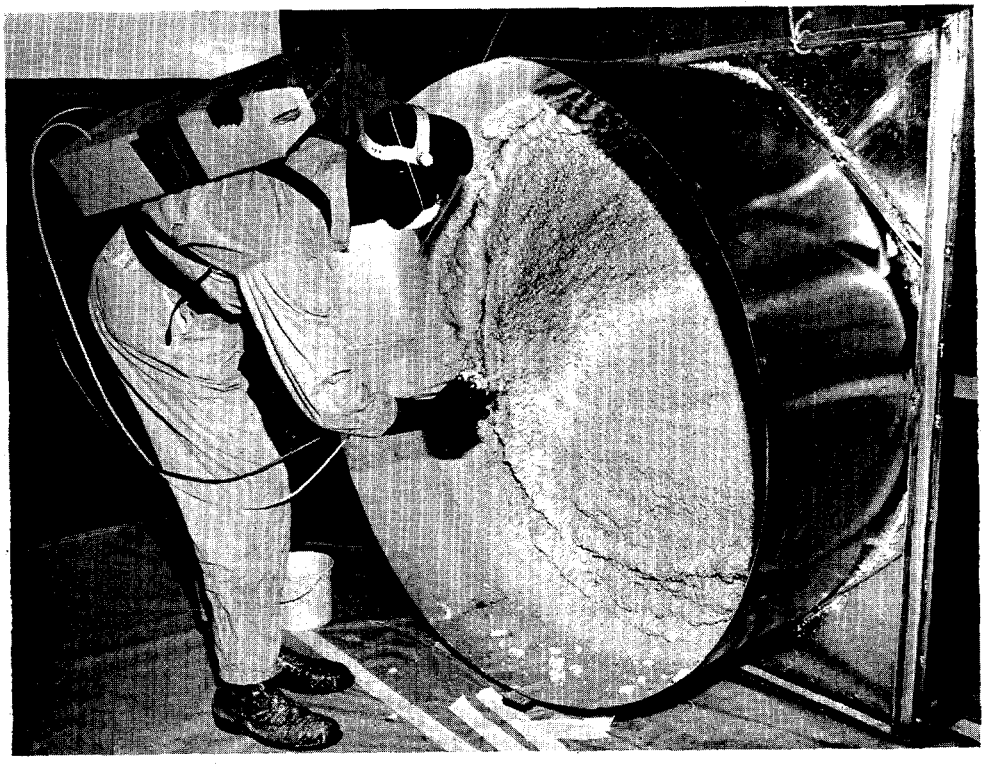

FIGURE 4. MOCKUP TEST OF URETHANE FOAM TO SEAL FILTER NOZZLES

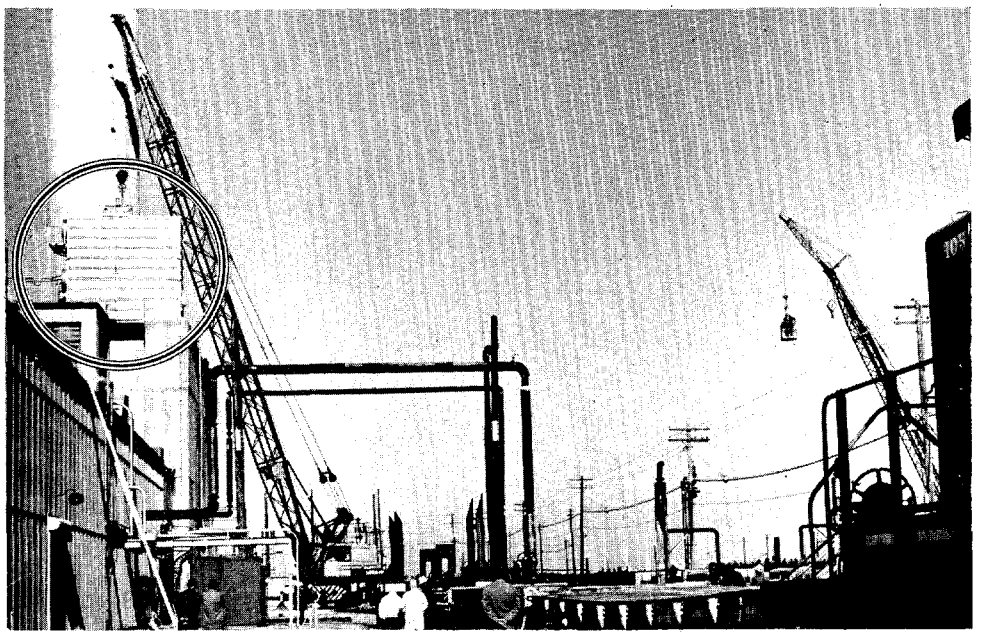

FIGURE 5. REMOVING CONTAMINATED FILTER COMPARTMENT FROM ROOF OF K REACTOR BUILDING

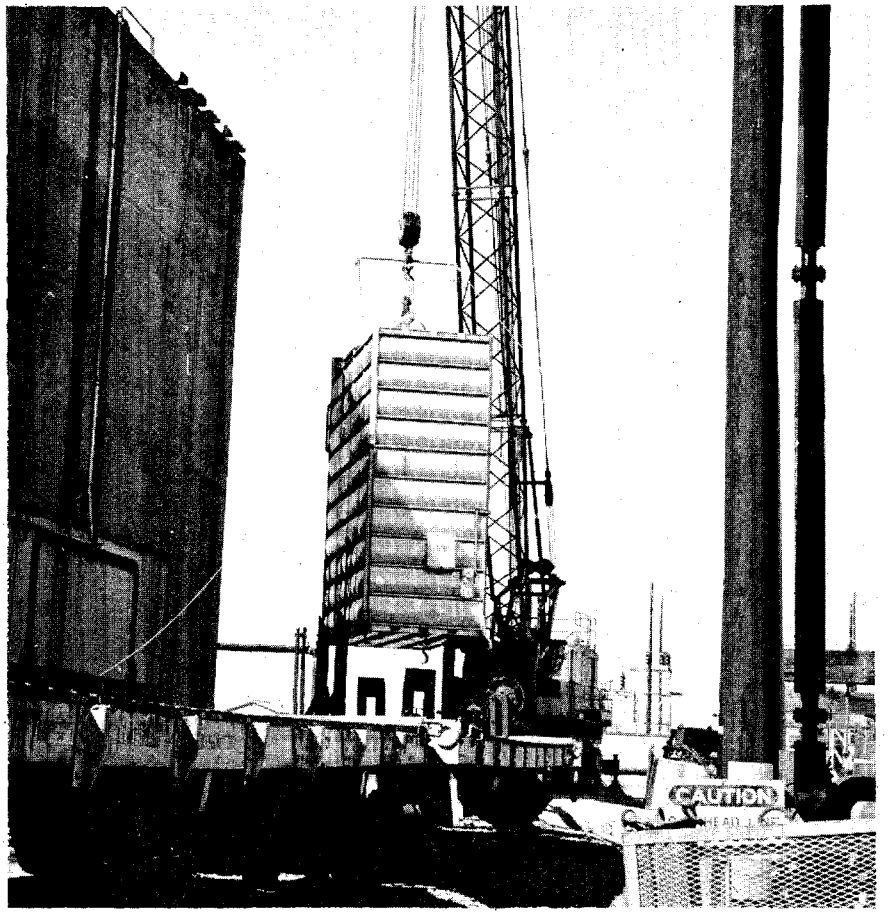

FIGURE 6. PLACING CONTAMINATED FILTER COMPARTMENT ON MODIFIED RAILROAD CAR

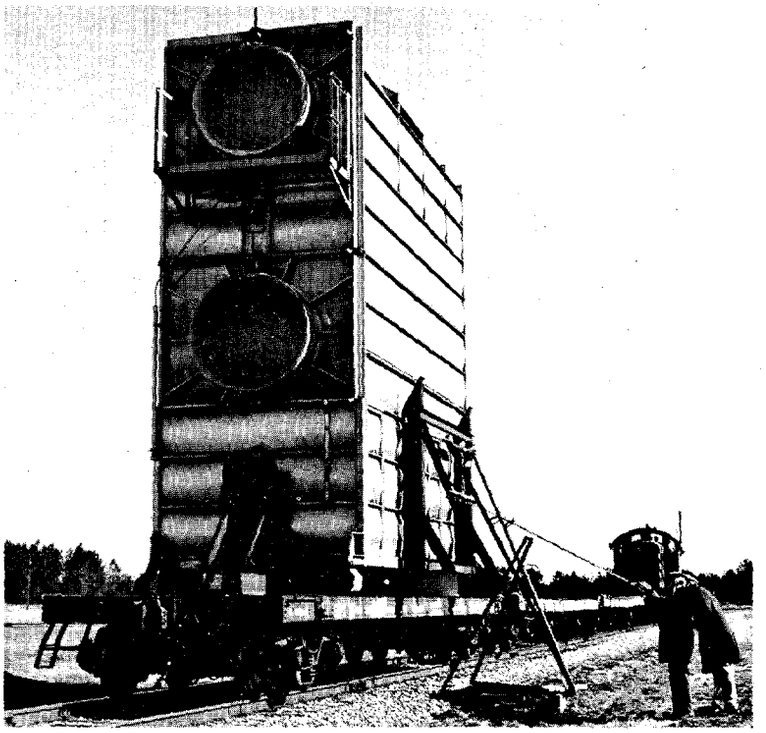

FIGURE 7. TRAIN WITH CONTAMINATED FILTER COMPARTMENT EN ROUTE TO STORAGE AREA.

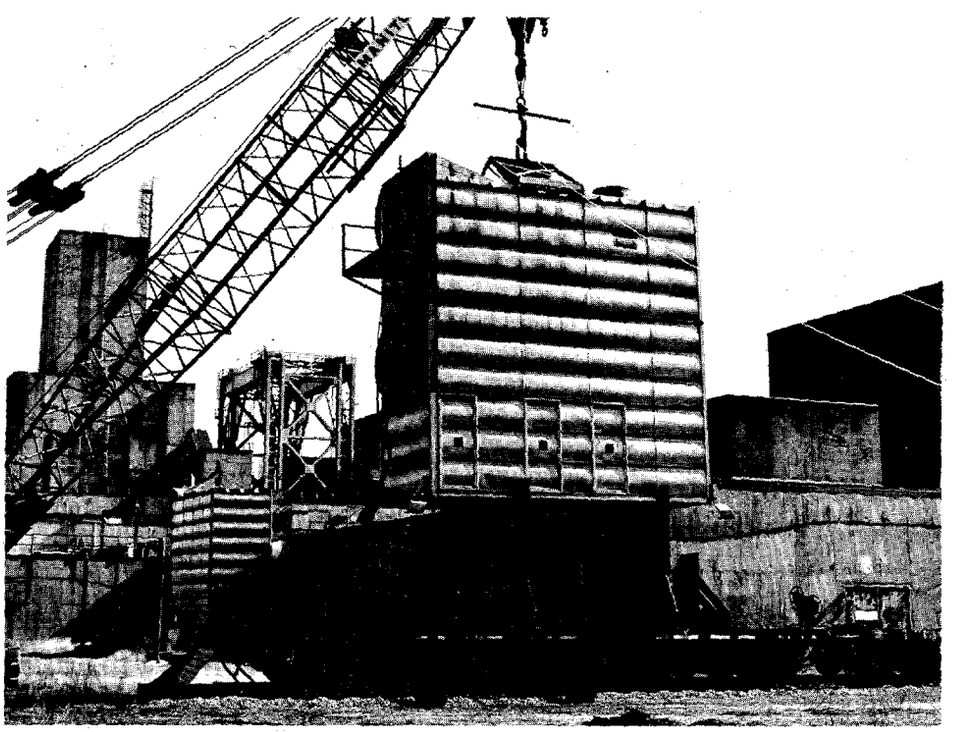

FIGURE 8. CONTAMINATED COMPARTMENTS DELIVERED TO STORAGE.

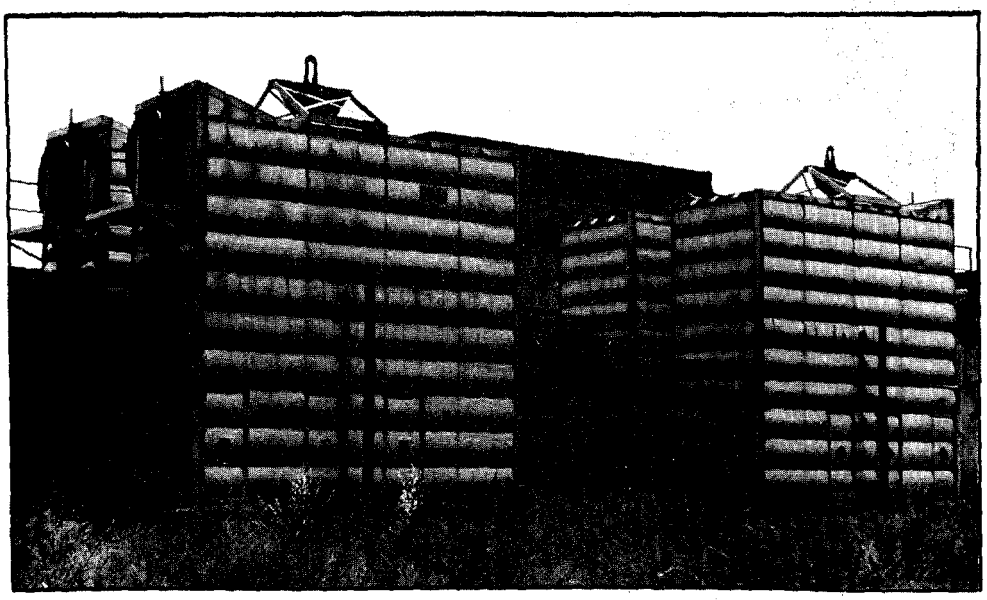

FIGURE 9. CONTAMINATED COMPARTMENTS IN STORAGE

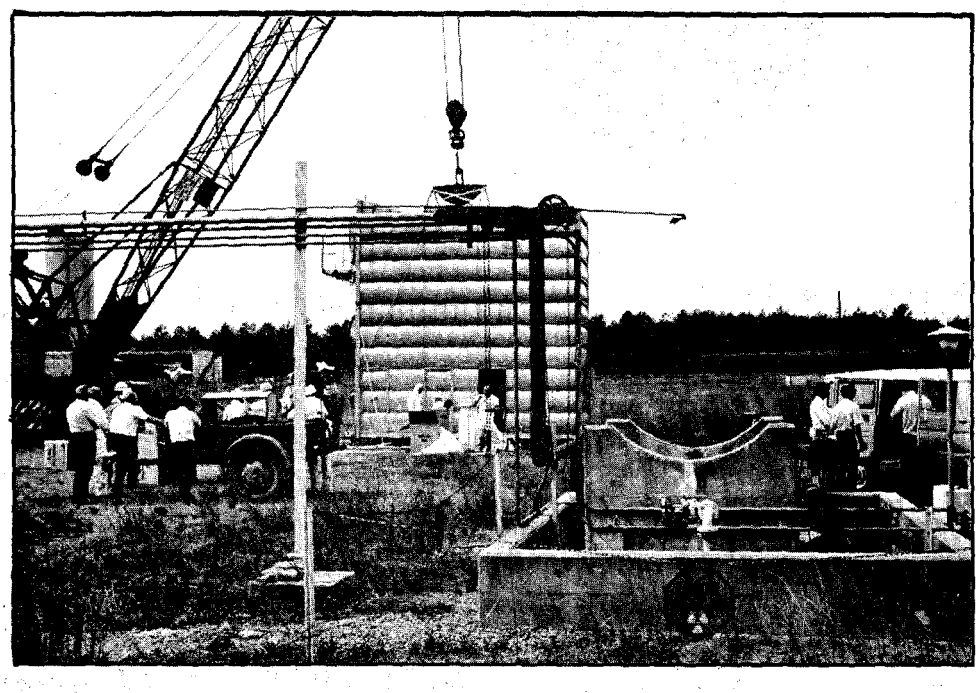

FIGURE 10. INITIAL ENTRY INTO CONTAMINATED FILTER COMPARTMENTS

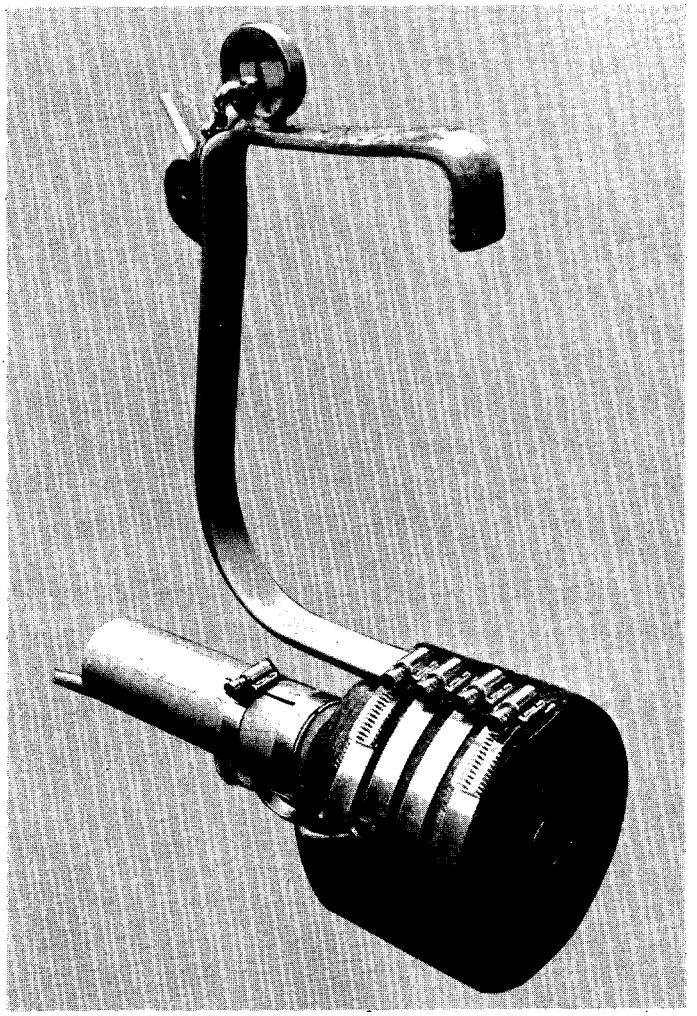

FIGURE 11. COLLIMATED PROBE ASSEMBLY

FIGURE 12. RADIATION MEASUREMENTS IN "CONSTANT GEOMETRY" FIXTURE

(-26) -30	-6	+4	(+11) -5
-9	(+23) +18	+17	+4
-19	+12	(+4) +16	+7
(-14) -20	+2	+12	+6
-15	(-2) +7	+9	+6
-13	+2	(-2) +10	(+11) +8
(-14) -14	+3	+11	+3
-20	-3	(-2) +1	(+11) -4

(Looking from Downstream Side)

*FIGURE 13. COMPARTMENT K-6

-3	-2	0	+1
-1	+2	0	-3
+1	+2	+1	+1
0	+2	+4	0
0	+4	+1	-1
-1	+2	+1	-2
-2	+1	0	+1
-4	-1	-2	-1

(Looking from Downstream Side)

FIGURE 14. COMPARTMENT K-3

Distribution of activity on particulate filters, % deviation from mean.
Viewed from downstream side.

*Data in parentheses are from measurements on selected filters in "constant geometry" fixture.
Other data are from shielded probe measurements on filters in the compartment.

DISCUSSION

ZAVADOSKI: I am wondering how you measured or determined the quantity that was released, i.e., the 3 millicuries that you mentioned?

LITTLE: We have normal health physics continuous monitoring equipment in the stack of the building, following the filter compartments. A sample stream is passed continuously through a fiberglass filter paper and the filter is counted once a week. A count immediately after the accident indicated a 3 millicurie release.

HIGH CAPACITY DOP AEROSOL GENERATOR*
Progress Report

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Abstract

The objective of this program was to develop a portable DOP** aerosol generator that would produce sufficient aerosol at a constant rate for the period of time required to test high capacity HEPA filter systems at rated flow. Such a generator would make it possible to measure the efficiency quickly and quantitatively. Also, with suitable access to the ventilation system, tests could be performed remotely, thus avoiding radiation exposure to personnel conducting the tests.

An experimental model has been designed, built, and successfully used at Hanford since October, 1971, to test filter systems which have rated flows up to 125,000 cfm. Based on experience with the unit, filter systems with capacities up to 200,000 cfm can be tested. This report describes the experimental unit and discusses the future objectives.

Introduction

The function of a DOP generator is to convert liquid DOP into an aerosol of finely divided spherical droplets, most of which are less than one micron in diameter. The aerosol is introduced upstream of the test filter bank and thoroughly mixed with the exhaust ventilation air. The concentrations upstream and downstream of the filters are measured by forward light scattering and the collection efficiency is determined therefrom.

On the basis of photometer sensitivity, it is suggested that the upstream concentration of DOP be no less than 20 micrograms per liter of air[1]. Thus, the quantity of aerosol produced by a generator determines the maximum flow at which a filter system can be tested. Generators are commercially available, but none produce the quantity of aerosol required to test large filter systems.

Based on past experience in the design and operation of DOP penetrometers and portable generators, an attempt has been made to develop a high capacity portable generator suitable for in-place testing large HEPA confinement systems at rated flow. Although high capacity was the major objective, consideration was also given to the possibility of producing a monodisperse 0.3 micron aerosol.

Concept

The concept of aerosol generation applied here is to heat DOP beyond its boiling point in an inert atmosphere, quench the evolved vapor with an inert gas

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* Work performed under USAEC Contract AT(45-1)-1837.

** Di-octyl phthalate.

stream and immediately release the aerosol to the atmosphere. The temperature of the vapor before quenching is 390 ± 5°C.* The purpose of the high operating temperature is to achieve rapid vaporization. The purpose in quenching is two-fold: 1) to condense the vapor, and, 2) to reduce the aerosol temperature below the flash point of DOP, which is 216°C, before discharging to the atmosphere. The purpose of the inert gas is to prevent combustion and to limit chemical change to only those effects that elevated temperature may have on DOP.

Description

As shown in a schematic flow diagram of the generator system, Figure 1, N_2 from the right cylinder passes through a pressure reducing valve (PRV) and divides into two streams. One stream passes through the gas flow meter, the other pressurizes the DOP reservoir forcing the liquid through its flow meter. The two streams rejoin to form a mixture of DOP and N_2 which enters at the bottom of the heat exchanger. The vaporized DOP is quenched at the heat exchanger outlet, with N_2 from the left bank of cylinders.

Figure 2 shows a cross-section view of the heat exchanger. DOP and N_2 enter at the bottom left, pass through the annular space between the cartridge element and the barrel where the DOP is vaporized. Upon leaving the heating section it is quenched by the N_2 stream entering at top left.

Figure 3 shows the heat exchanger before applying the insulation. Subsequent to taking this photograph, the tee on top of the assembly was replaced with a cross tee. Quenching N_2 enters the cross tee opposite the outlet. Also, fittings marked X-1, X-2, and X-3 are not currently used in the experimental model.

The heat exchanger barrel is made from brass bar, 2 x 2 x 30 inches. The barrel inside diameter is about 1/8 inch larger than the cartridge element. A 1000-watt, 240-volt strip element is attached to each side of the barrel. The cartridge element is 1.287 inches O.D., 24 inches long, 1780 watts, 240 volts. It is inserted through the junction box as shown in Figure 4 and is secured at the bottom by a packing gland. Four spacers (not shown) hold the top of the cartridge element centered in the barrel. Two thermocouples (TC) operate indicating temperature controllers, a third TC operates a temperature indicator.

The generator requires a 30-amp, 220-volt, single phase power supply. A common hookup in the field is a 30-amp disconnect tied into a nearby lighting panel. This connects to an auto-transformer which delivers 240 volts to the heating elements as called for by the temperature controllers. One controller regulates current to the four strip elements, another controller regulates current to the cartridge element. The maximum current demand is 24.1 amps at 240 volts.

Operation

After a warmup period of about 40 minutes, the generator has reached its operating temperature and is ready for operation. When operated at its present maximum capacity, the flow rate of mixture into the heat exchanger is 170 ml DOP

* DOP B.P. = 384°C.

and 1 ft^3 N_2 per minute. The N_2 quench flow is about 20 cfm. The mixture can be reduced to any desired flow, accompanied by a proportional reduction in N_2 quench flow. With a two-gallon DOP reservoir, full capacity operation can continue at least 15 minutes. With proper mixing of aerosol and ventilation air, the variation in upstream concentration holds within ± 2 percent during the test. The relocation of one of the controlling TC's may reduce this variation.

Gravimetric sampling of aerosol at the upstream face of a 125,000 cfm exhaust filter bank operating at full flow indicated a concentration of 32 micrograms of DOP per liter of air.

Particle Size

Particle size measurements were made using the procedures described by J. K. Thompson[2]. The jet impactors were loaned by Naval Research Laboratory, Washington, D.C. The apparatus arrangement, as shown in Figure 5 was identical to that described by Thompson except for the adjustable hose clamp and pressure gage. The clamp was used on the sample line leading to the light scattering chamber (LSC) in order to maintain a constant LSC pressure for all sampling situations. The pressure was adjusted to -11.5 inches w.g. at the LSC. Without this adjustment, the LSC pressure would have ranged from -0.2 to -11.5 inches w.g. depending on the pressure drop of the jet impactor used.

A portion of a reactor exhaust ventilation system was used for the study. Figure 6 shows the general view of the generating system, with the aerosol being introduced upstream of the fan. Figure 7 shows a close-up of the generator in operation. Not shown is the sampling location at the filter cell, which was about 200 feet downstream from this point.

The jet impactor-light scattering measurements are shown as a log probability plot in Figure 8. These data indicate a weighted geometric mean diameter of 0.64 micron.

Future Objectives

Future objectives for the generator call for the development of better control of the contact between the vapor stream and the quenching N_2. This should increase the efficiency of quenching which may also have a beneficial effect on particle size.

A liquified petroleum gas-fired heat exchanger has been designed and built and will soon be tested. It has a higher potential capacity than the electrically powered model. It uses a readily available portable fuel which could simplify power supply problems.

Investigations will be made to determine if a more desirable size aerosol can be achieved with either generator.

Minor attention has been given to the size and weight of the working model; however, these factors will be given full consideration in the development of a prototype.

Conclusion

1. An experimental high capacity DOP aerosol generator has been designed, built, and tested. It will produce sufficient aerosol at a constant rate for the period of time required to quantitatively test HEPA filter systems up to 200,000 cfm capacity.

2. Before developing the working model into a prototype, the gas-fired unit should be evaluated.

References

1. W. L. Anderson, personal communication.

2. J. K. Thompson, "Determination of Aerosol Size Distributions by Jet Impactor-Light Scattering Technique," Anal Chem, Vol. 29, No. 12, Dec. 1957, pp.1847-1850.

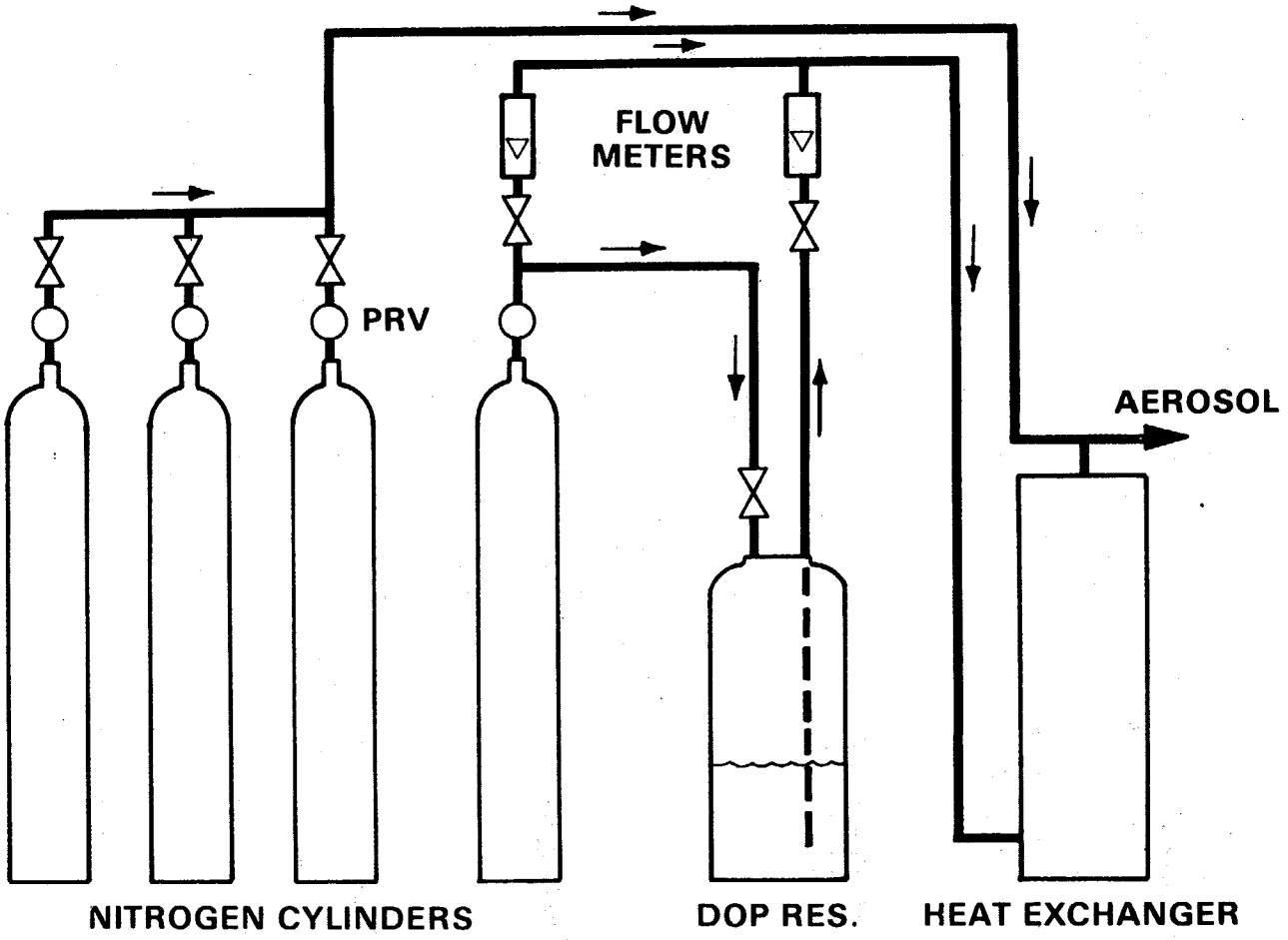

Figure 1. Schematic Flow Diagram of DOP Aerosol Generating System.

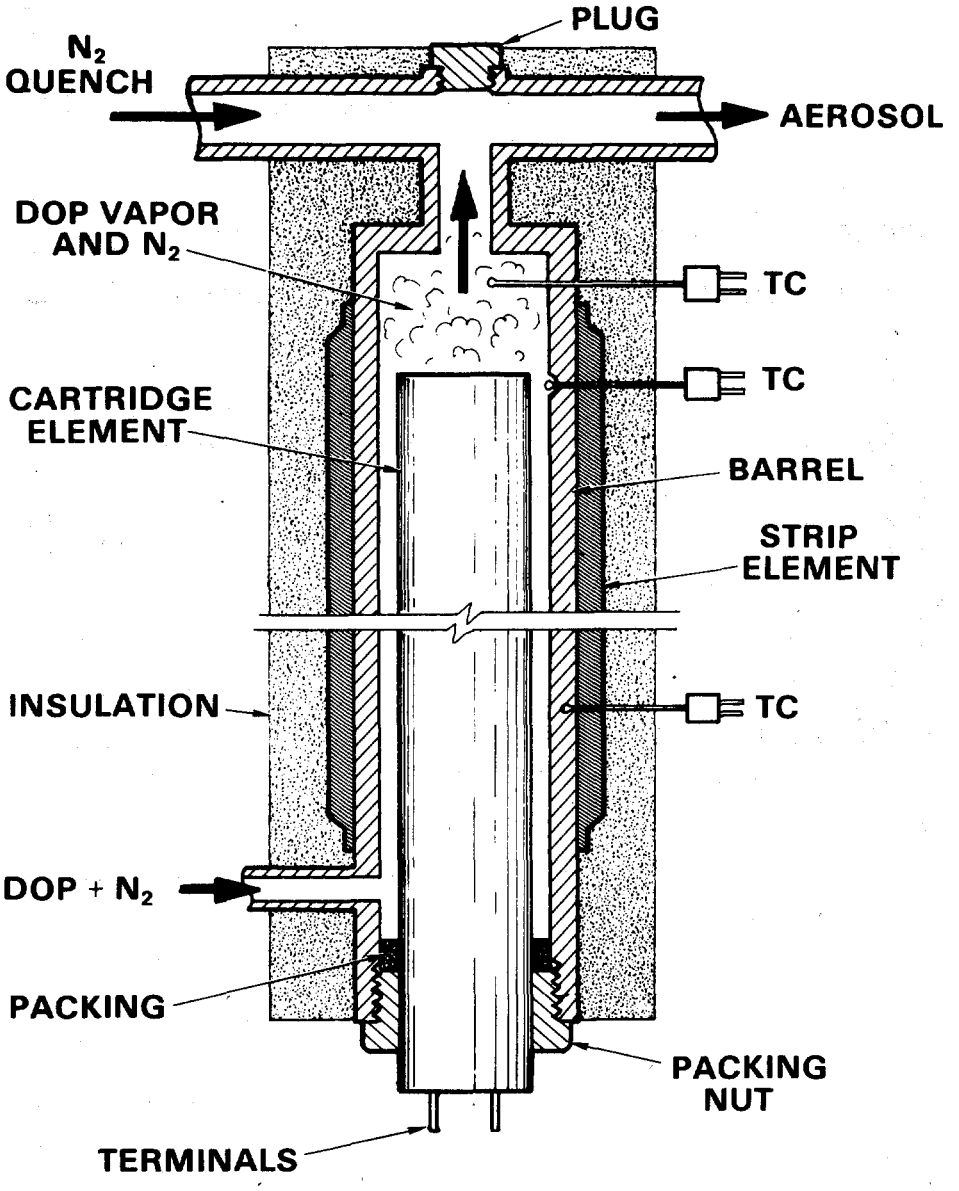

Figure 2. Schematic Cross Section of DOP Heat Exchanger

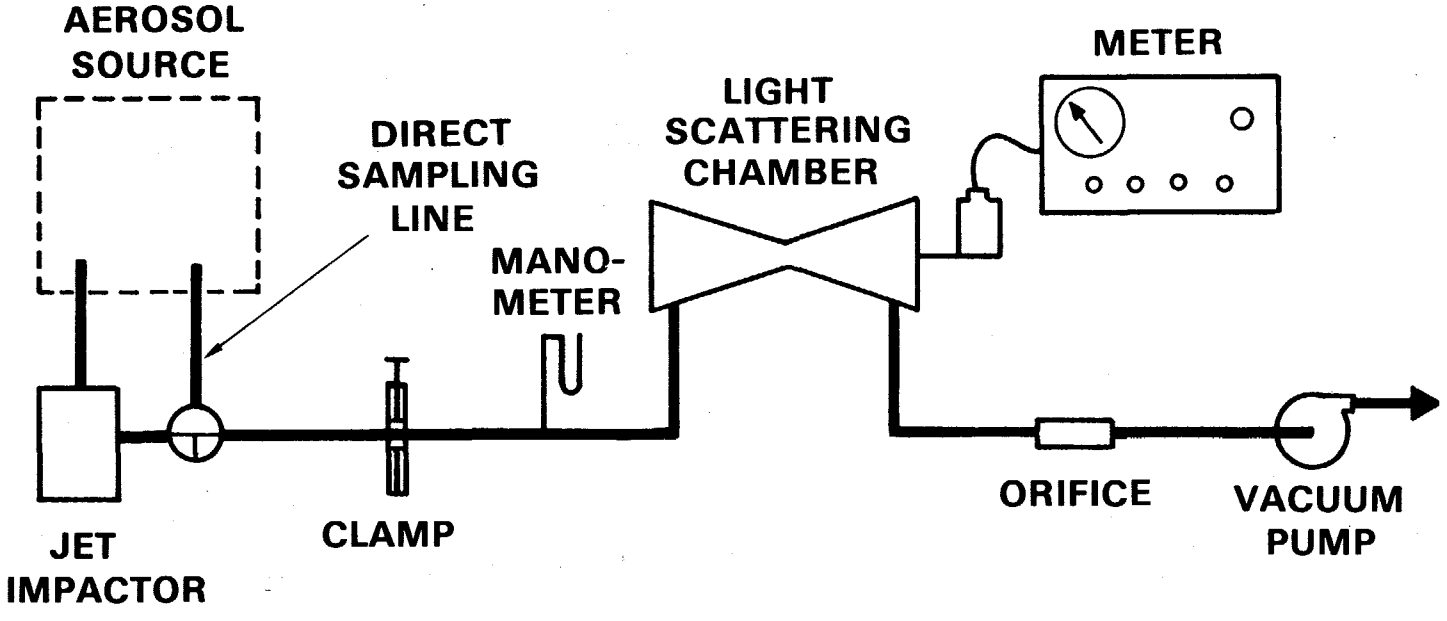

Figure 5. Arrangement of Apparatus for Particle Size Measurements

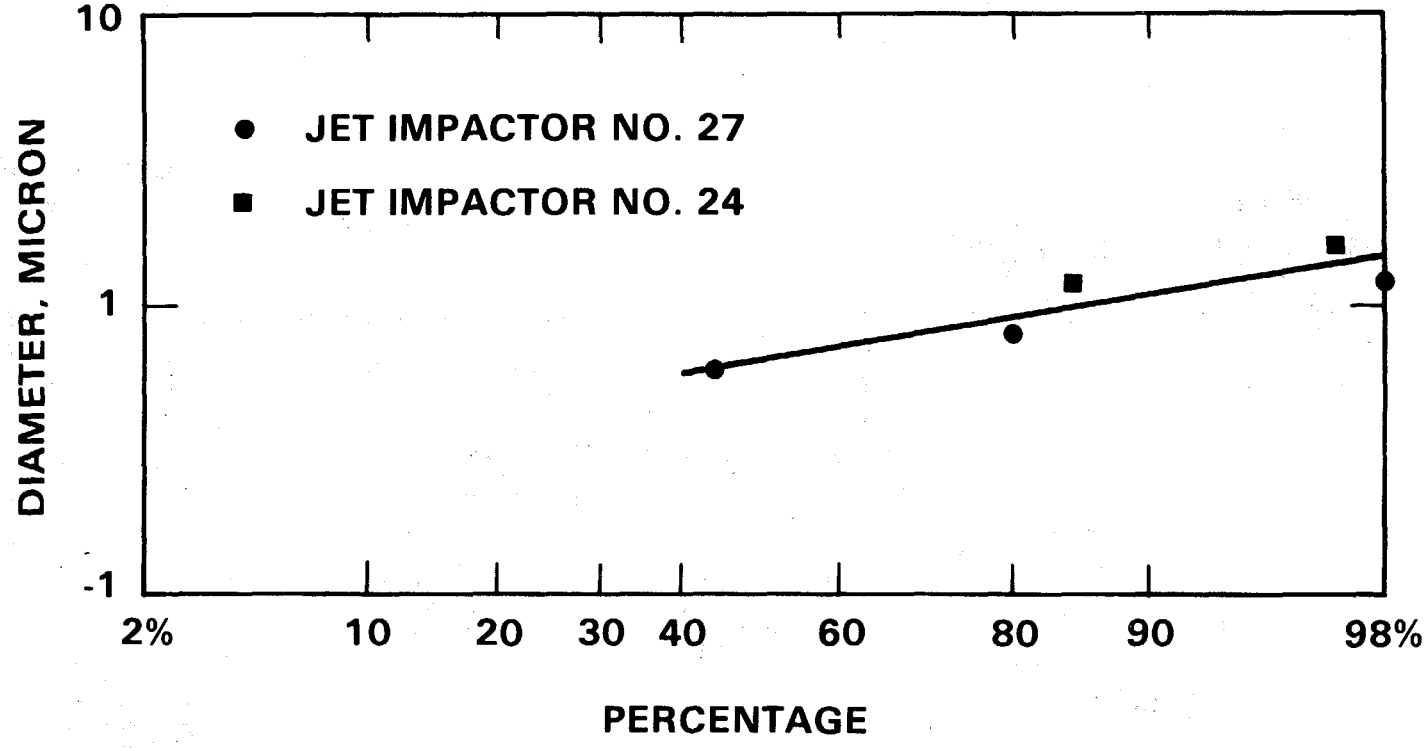

Figure 8. Jet Impactor - Light Scattering Measurements

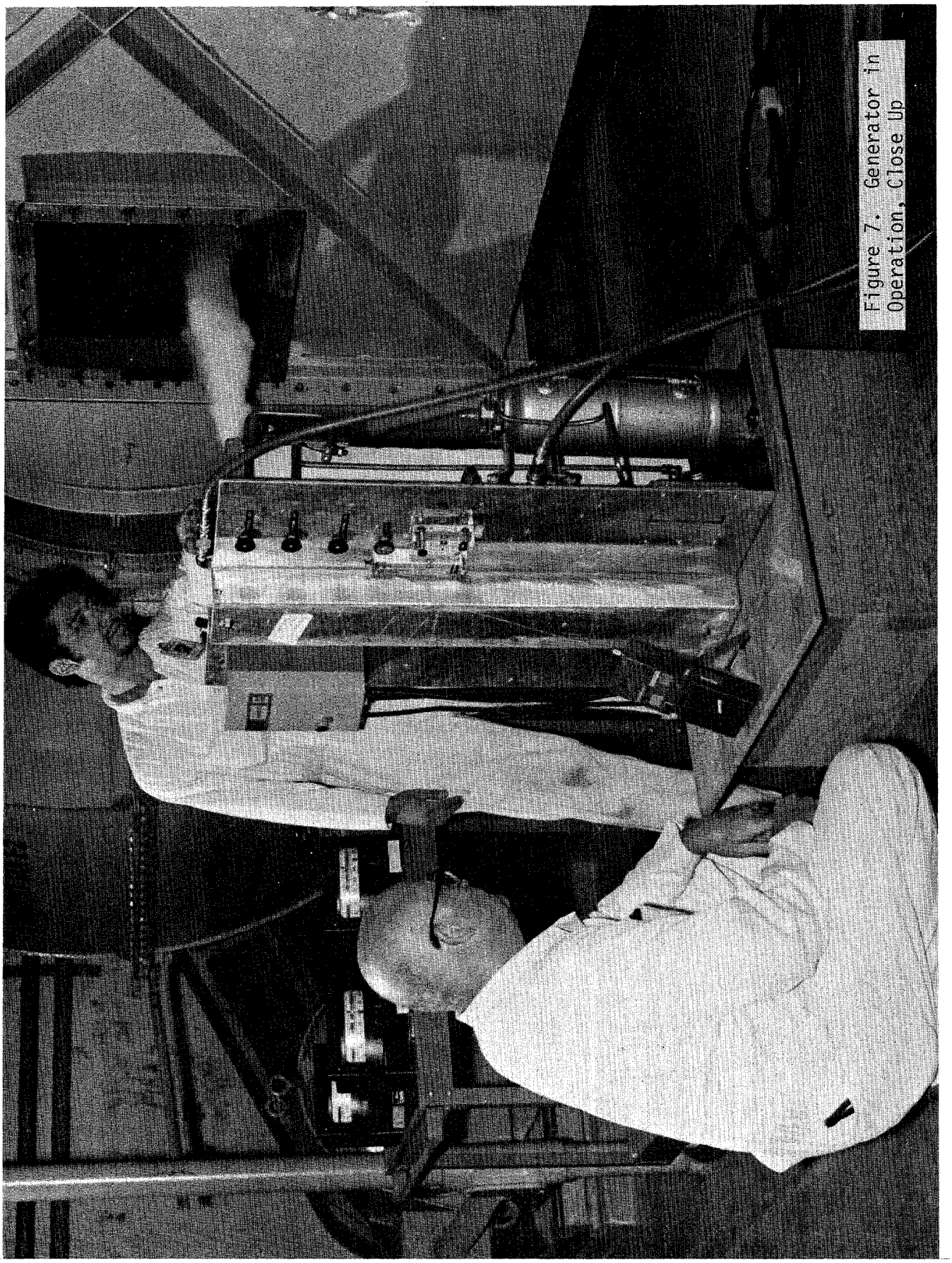

Figure 7. Generator in Operation, Close Up

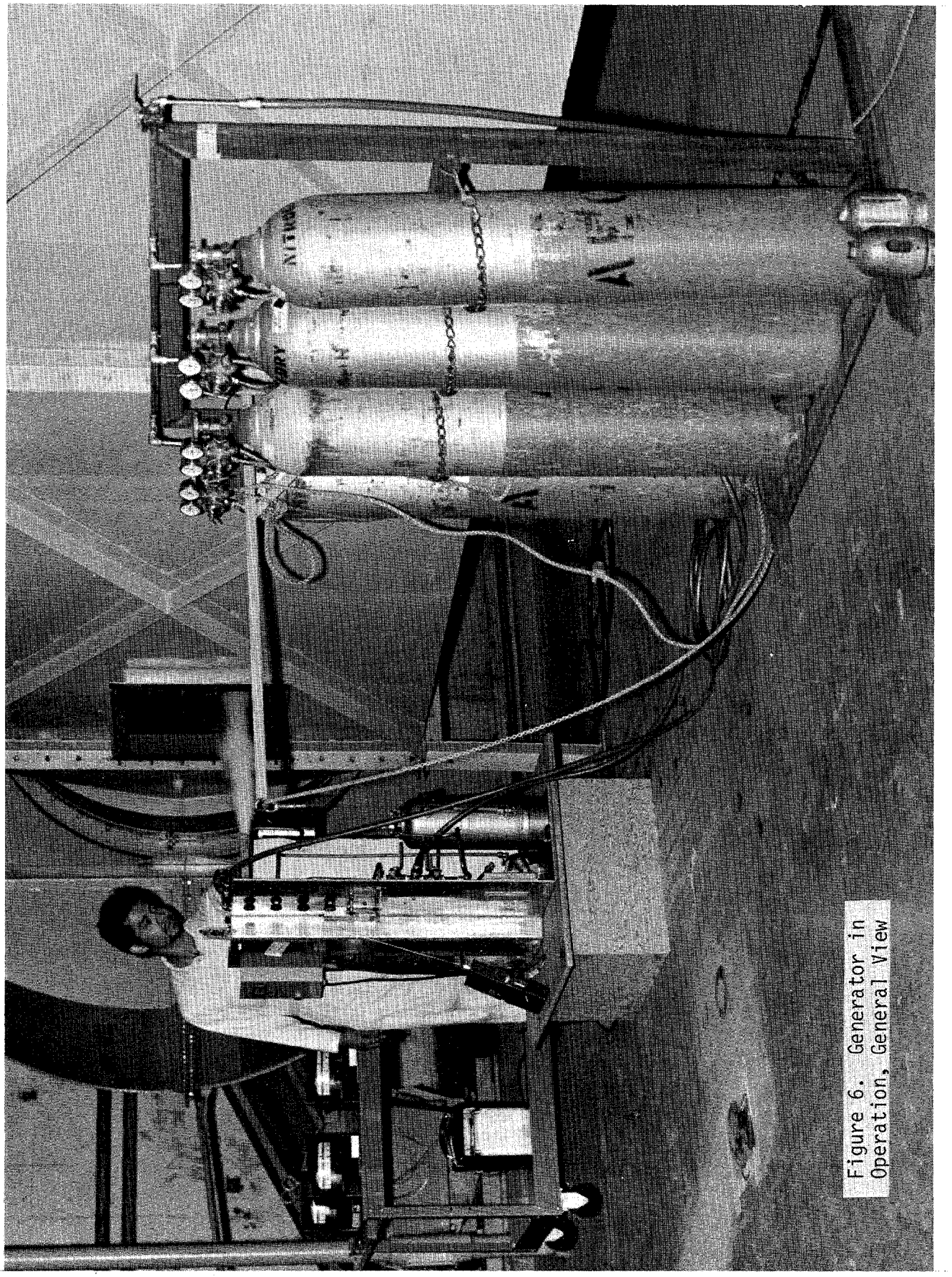

Figure 6. Generator in Operation, General View

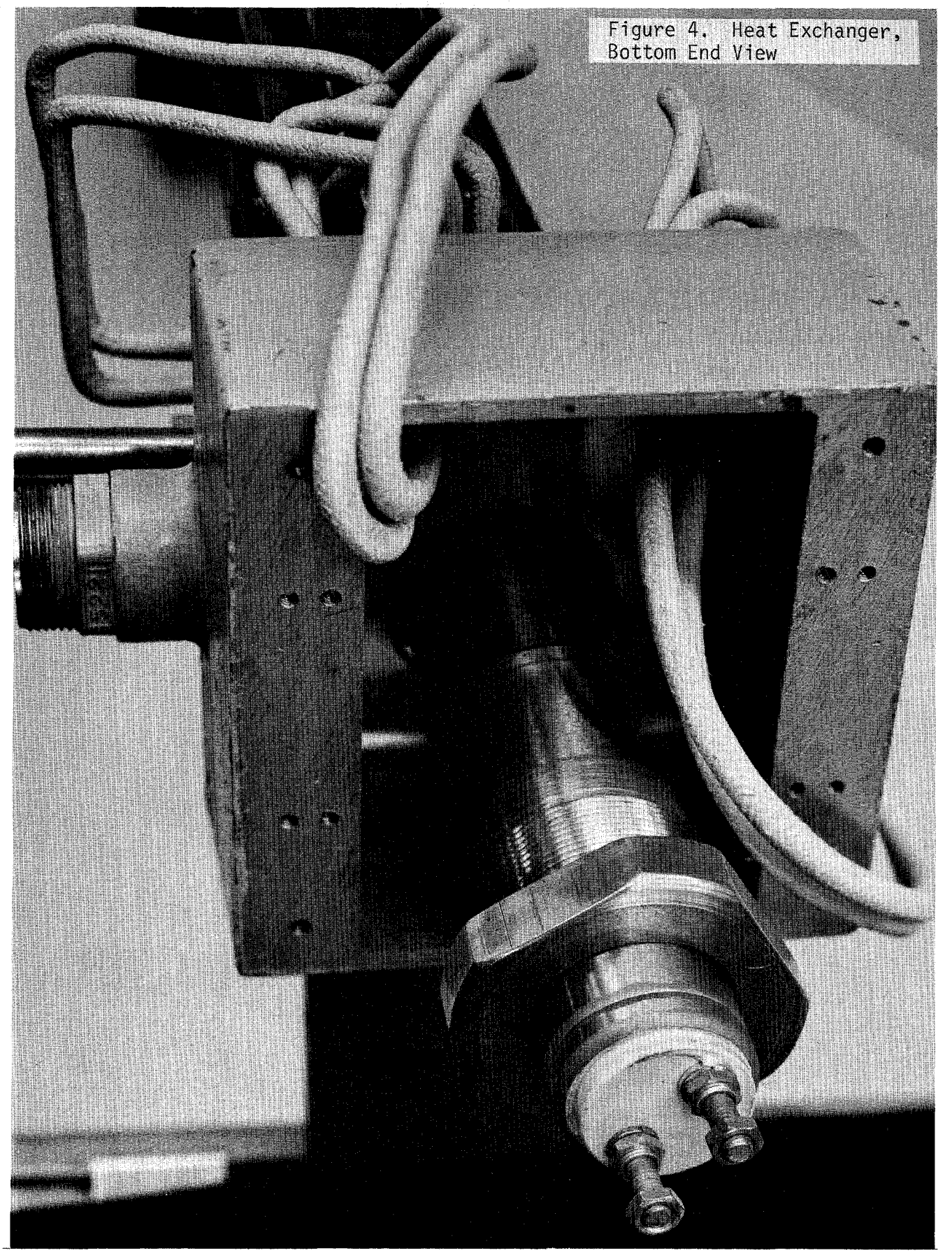

Figure 4. Heat Exchanger, Bottom End View

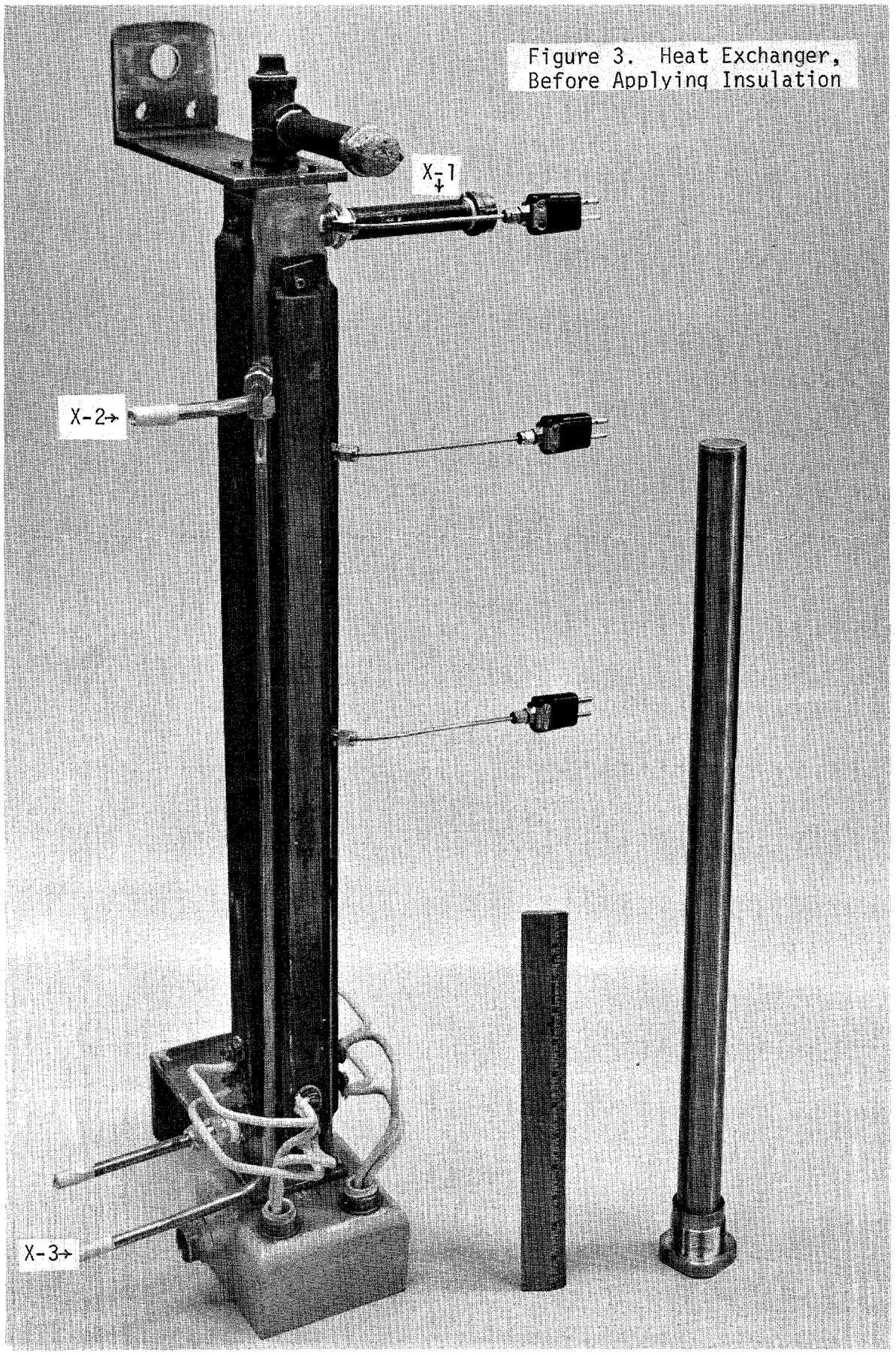

Figure 3. Heat Exchanger, Before Applying Insulation

DISCUSSION

GOULET: Is there any danger of reaching explosive concentrations when mixing the aerosol with air?

ROLIE: Yes, but we didn't try that out. We decided, from the beginning, to use nitrogen.

GOULET: When you mix it with air, you get oxygen mixed with the aerosol, do you not?

ROLIE: This is true, but the temperature of the aerosol, as it exits from the generator, is lower than its flash point of 216°C. This, of course, is one of the criteria used in the design of the generator; i.e., to get the exit temperature below the flash point.

GOVERNMENT-INDUSTRY MEETING ON FILTERS, MEDIA, AND MEDIA TESTING

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Many of the accomplishments of the AEC air filtration program achieved thus far have been due to the efforts of an informed working group concerned with high efficiency filters. The existence of this group has now spanned five air cleaning conferences and has shown successive growth and participation at each one. At the 10th Conference in New York, 32 people comprised the working group, thus necessitating a formal organizational structure and a prepared agenda of five separate discussion items. At the 11th Conference in Hanford, 42 persons were in attendance at the working sessions and eight discussion subjects were resolved. At this, the 12th Conference, 48 persons comprised the assembled body; 28 from industry, 8 from Government, 7 contract investigators and 3 international guests. Many other individuals and/or companies had expressed a desire to participate but the restriction of the attendees was considered to be mandatory in order to keep the active discussions under rational control.

The most recent session of this working group was held this past Monday morning and was devoted to a series of discussions on subjects of current interest. This session, following the precedent of earlier meetings, related the operating requirements placed on the high efficiency filter and the capacity of industry to meet them. To this end, the collected talents of the assembled body were unified toward the solution of the problems of the particulate filter, its components, and methods of test. Representatives of all of the facets of the industrial complex were present, from the basic fiber suppliers, through the media producers, and finally to the filter unit fabricators. Research organizations from R&D government laboratories and academic institutions contributed status reports on work currently underway. Users at various levels expressed their problems and actively participated in the discussion.

The following review of the discussions may seem to be an agglomeration of information and show little continuity of thought. It is my intent to review for you, in abstract form, the items of committee deliberation. I will address the items in the order of their discussion. Under the whip cracking of our chairman and without the smoke screen that his cigar usually generates, we completed all agenda items in record time.

At the recent session, 11 separate subjects were discussed. These are:

1. Filter Media Caliper: Continued surveillance of the HEPA filter materials indicates that some of the inspected media does not meet specification requirements, particularly with respect to media caliper (thickness). Even though a uniform test method (TAPPI T-411) has been specified, sufficient deviations in test equipment and/or personnel techniques have resulted in erroneous measurements and sub-specification material. This deficiency was also recognized at the 11th Air Cleaning Conference and remedial action recommended.

Based upon an examination of the standard methods of test (ASTM and TAPPI), a survey of the instrumentation available for the precision measurement, and a determination of the present methodology of the industrial community, the Testing Machine bench micrometer (TMI 549-M) was approved as the method for caliper determination of the HEPA filter media. Experimentation was conducted to document the various previous methods of test and to correlate the new method with earlier devices and techniques. The procedures of TAPPI 411 will continue as the proper technique for procedures and calibration; TAPPI 400 will continue as the criteria for sample collection. No changes in MIL SPEC F-51079A for HEPA media with respect to minimum caliper thickness are either recommended or contemplated.

2. Determination of % Combustibles: Mil Specification F-51079A requires that % combustion tests be conducted over the procedures of TAPPI T-413. This test originally called for the heating of the media to 925 ± 25°C for at least 3 hours before final determinations could be made. A recent change in T-413 however, has established the heating criteria as 1000 ± 25°C until constant weight is achieved. It should be recognized that the procedures specified were devised for materials of high organic content and very low ash; hence the long time required to reach equilibrium. Since present fiberglass HEPA materials represent the opposite extreme (very high residual and low organic) the specified test procedures are considered to be too rigid and unrealistic and modifications to the standard method were deemed necessary. Based upon extensive experimental data it was recommended that a modification of the procedures of T-413 be adopted for the determination of % combustibles in HEPA filter media. Either 800°C for 2 hours or 1000°C for 5 minutes can be specified as the temperature-time relationship. Since both methods give comparable results the choice of test is left to the discretion of the media supplier; the choice would obviously be a trade-off which would permit the performance of the test with a minimum cost-time factor.

3. Contamination of Glass Fibers: Surveillance of HEPA filter materials has shown a definite trend toward the use of increasing amounts of organic binders as a means of increasing tensile strength and waterproofing of the completed media. This has been reflected by the increase in the % combustion values as indicated by the performance tests. When contacted, the media suppliers related that since there had been little change in either the materials or concentrations of the binders, the % combustion increase must be from some other source. One area of interest appeared to be the contamination level of the as received (AR) fibers from glass fiber suppliers. Experimentation has confirmed that there is a small amount of contamination in all fiber samples tested thus far. The fact that the higher % combustion exists in the lower fiber diameters leads to the conclusion that the contamination source is an atmospheric one and results from the removal of these materials from the production process air; i.e., the finer the fiber, the better the filter for these materials. Chemical analyses of the contaminant identifies it as mostly organic in composition and similar to common lubricants and fuels associated with production operations. It was concluded that a residual combustible content of the bulk AR fibers utilized in HEPA filter media does exist. However

since no increase of this % combustion has been observed as a function of the type of glass or date of manufacture, the % increase of the % combustibles in the final media cannot be attributed to the AR fibers. It was further reasoned that since the % combustibles contributed by the AR fibers is small, that no change be made in MIL F-50179A for HEPA media with respect to maximum allowable % combustion.

4. Test Standards: Present specifications for quality control and testing of filtration media are based upon the procedures of the ASTM and/or TAPPI standards. A recent proposed standard has been received from the British Paper and Board Makers Association. Preliminary review has revealed that there are several areas of disagreement either by method of test or by omission of requirement. A concentrated effort will be made to resolve the differences and to prepare common testing standards that can be utilized in both the American and European areas.

5. Irradiation Tests on HEPA Media: Savannah River (SRL) has been conducting a series of experiments to determine the effects of high levels of gamma radiation on the materials utilized in the HEPA filter units. Using a Colbalt 60 source with a field strength of 4.5×10^7 rad/hr; it has been determined that a dose of 1×10^8 rads gives a distinct separation of the radiation resistant filter media from the nonradiation resistant media. Since consideration has been given to the inclusion of a requirement for this type of exposure into the media specification, it would be necessary for the individual media suppliers to qualify their specific media. Since SRL is not in a position to serve as the ultimate qualification source, an independent industrial company was investigated as to their capability to perform the tests and to the comparability of their results with the SRL experiments. It was known that a food irradiator of comparable strength had been successfully utilized at Winsor Nuclear (WN), Windsor Locks, Conn. It is understood that WN has a 3 mev Van de Graff and that exposures of 10^8 rads were well within their capabilities. Since this source was readily available, a direct comparison of material effects was made between the SRL and WN exposure conditions. Based upon the results obtained to date, it has been determined that the exposures and the resultant material effects are the same for both systems. It was therefore recommended that the WN radiation facility be utilized for qualification testing of filter media at the 1×10^8 rad level. It is further recommended that the various filter media suppliers utilize this facility for the R&D efforts in the development of new or modified binders and/or water repellants.

6. Radiation Deterioration Tests: From the studies on materials effects resulting from radiation exposures at SRL, it had been determined that the parameters where damage was the most significant were the media waterproofing properties and the media tensile strength. All of the other properties monitored showed little, if any, change following irradiation. Since the tests used in this evaluation were unique and not covered by standard methods, new techniques were required. For the water repellancy and wet strength tests, a 2-inch unsupported test area was provided by inserting the media between two test flanges. A 6-inch column of water was then placed over the media and the

time (in seconds) measured for the first drop of water to emerge from the downstream face. This value has been stated as the water penetration time. Following appearance of this first water drop, the water column height was increased at the rate of 1-inch every 5 seconds until the media ruptured. The height of the water column required to rupture the media is taken as a measure of the wet strength. If the water head is less than the 6-inch specified in the water penetration test, this means that the paper saturated and broke before the specified head for water repellancy could be achieved. This phenomenon frequently occurs in those instances where the water repellancy has been dissipated by the irradiation. Based upon the tests to date, it is recommended that the aforementioned tests be adopted and that tentative media specification values be established for water penetration, wet strength and dry strength following exposures at 1×10^8 rads. These tentative values are:

Water Repellancy	>	100 secs
Wet Strength	>	10 in H_2O
Dry Strength	>	1.5 #/in

7. Media Deterioration Thru Use: SRL has reported that under normal use conditions, filter media has been found to deteriorate with respect to its water repellancy and tensile strength. It has been determined that this deterioration has resulted from an as yet unidentified air contaminant present in the air processing system. Rinsing the media with alcohol can restore the original water repellancy but has no effect on the decreased tensile strengths. In addition, the alcohol extracts when applied to new filter materials will destroy its water repellancy and reduce its tensile strength. It is postulated that an air contaminant is forming a monolayer or crystal layer on the surface complex and changing the original surface from hydrophobic to hydrophilic. Extraction of this material by solution restores the material to its original water repellant state. If the material is crystalline (nitrate) then recrystalizing on the new media would start the process all over again. In addition the crystalline material might form an acid product when combined with water; this acid could attack and/or destroy the binder materials in a nonrecoverable manner. Further work on the actual contaminant and its action mechanism will be conducted to clarify the deterioration process.

8. Filter Specifications: Specifications for both the media and filter units are under review and/or consideration for change. Specific areas to be addressed are the heated air tests, material specifications following irradiation at 1×10^8 rads, and specific physical tests related to media flexing and or folding response. The present mandrel tests for the latter requirement will be examined for their adequacy in forecasting the media performance in the completed filter unit. Minor revisions and editorial changes will likewise be accomplished in the revised specification. No major changes in present performance criteria are anticipated.

9. QPL Tests: After seven years of discussions and arbitration it now appears

that the Army Chemical Corps (ACC) is ready to proceed with qualification products listings for HEPA filtration units. It is anticipated that such testing will be possible in the next 6 months with subsequent listing of the qualified units. The actual cost of such tests have not been established as yet but in any event will be borne by the filter unit fabricators.

10. Smoke Penetrometer Correlation: A preliminary attempt by the AEC in 1970 to correlate the smoke penetration meters utilized by its various laboratories and test stations met with only partial success. Differences in test conditions and failure of several of the test components resulted in only a portion of the original objectives being satisfied. A more comprehensive round-robin correlation program, conducted in the blind mode, has just been completed. A total of nine different laboratories participated in the testing program. It is gratifying to know that 7 out of the 9 were in complete agreement on the first attempt with respect to the media resistance determinations. Subsequent rechecks at the installations where initial agreement was not achieved showed their original measurements to be in error; their redetermined measurements were in line with the expected values. Correlation of the particulate penetrations were also in close agreement. It is believed that small differences (1 mm resistance or .001% penetration) resulted from failure to accurately adjust the flow conditions with the calibrated resistance plate. This is believed to be especially true with those organizations that do not use the plate at all. It was concluded that if proper attention is given to the particle size adjustment and if the calibrated plate is utilized for establishing the flow, penetration calibration can be achieved and the determined results meaningful. It was recommended that a new round-robin series of tests be conducted to maintain the established correlation.

11. Fluoride Resistant Glass Media: Certain production processes result in the requirement for particulate filtration of large quantities of air that also contain fluorides. This type of material causes accelerated deterioration of the glass fiber filters used for the clean-up of the off-gas process air. This creates a logistic and economic problem due to the frequent replacement required and a potentially health and safety problem due to the unpredictable failure rates of the filter units. Preliminary screening of replacement filter media by Oak Ridge has identified a material composed of a specially formulated glass fiber (L-134) and delaminated crocidolite asbestos as being especially corrosion resistant and suitable for substitution in the filter units currently used. These tests were conducted on media specially prepared in the laboratory and only after efforts to scale up to full production capability was it realized that certain inherent problems existed in both the manufacture of the L-134 fiber and its incorporation into a suitable media for filter unit fabrication. L-134 is produced by Johns-Manville over a special proprietary glass melt formulation. During the original production of the fiber a major obstacle appeared in the quality of the fiber produced. Fibers manufactured in the early production process contained a high percentage of "shot" which seemed to be inherent in the process itself. Samples of this L-134 fiber contained about 50% "shot" that appeared in the fiber mats as either tadpoles or dumbells. The average fiber diameter was in the desired 5 to 6 micron range but the fiber appeared to

be very fragile and subject to fracture with even minimum processing. Efforts to adequately clean the fiber were not successful without degradation of the fiber itself. Average fiber lengths following the cleaning operations were of the order of 1/32 inch. In spite of the acknowledged deficiencies of the L-134 fiber, HERTY Foundation was successful in preparing handsheets and pilot mill production quantities of 2/3 L-134 and 1/3 asbestos media. These materials were found to be acceptable with respect to media specification and confirmatory tests at Oak Ridge verified its corrosion resistance. Sufficient media was produced to permit the fabrication of several 6 x 6 glove box filter units. Thus unit fabrication equipment could be evaluated with respect to its ability to process the new media. After fabrication the filter units were DOP penetration tested and found to be unacceptable. Even though the paper conformed to the media specifications, the unit penetration was determined to be in excess of 1%. Careful examination showed that the media was delaminating (separating) at the fold ends and on occasion, small holes developing in these areas. Delamination occurs on both the sides of the media, with perhaps the top-side of the media more susceptible than the wire side. Careful examination of the media showed several layers of laminates and at times definite striations. This effect results from flocked rather than dispersed deposition of the fibers. Separator scuffs and/or punctures were also observed indicating that the media tensile strength was not sufficient to resist the man-handling of the pleating process. In general, the conclusion at this point seemed to be that the fiber cleaning process (shot removal) deteriorated the L-134 fiber to such an extent that unit production was not feasible. JM was asked to re-examine the fiber production process to determine if fibers of lower shot and longer length were possible. Using somewhat modified fiber forming equipment, a much improved fiber has been produced. JM has stated that the melt formulation is identical to that previously furnished and thus one would expect similar corrosion resistance. Examination of these fibers show them to have certain desirable features over those supplied earlier. Shot content has been reduced from the previous 50% down to the 8 to 10% level. Fiber lengths in the as received condition are considerably longer and lengths in excess of 25 mm are commonplace. It is concluded that the new L-134 fiber is the best by far of any received to date; the physical properties of this new L-134 fiber should overcome the previous stated deficiencies and permit acceptable filter unit fabrication. It was recommended that any shot removal processes be selected so that fiber treatment be as gentle as possible; in spite of its increased length, the fiber remains extremely brittle and subject to fracture. If at all possible transfer gear pumps should be avoided and devices utilizing acute turbulent flow conditions used with discretion. Tests on the new fiber have been conducted at OR and the media has been found to have equivalent, if not superior, fluoride corrosion resistivity. Roll stock from the new fiber has been made by Herty and several glove box sized filter units are currently under construction.

In conclusion, it should be reemphasized that this informal working group, with its diversified representation, provides a means for a comprehensive and expedient solution to the problems of the filtration industry. The total effort has proven invaluable because it permits the surfacing and

exposure of problems that might otherwise be lost in the quagmire of bureaucracy and management. The meetings are intended to be and actually are a working level distribution of data and expertise as well as a progress report of ongoing projects in the particle filtration areas. To this end, we feel that we have been successful and future sessions are comtemplated.