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PROTECTIVE EQUIPMENT EVALUATION PROGRAM

QUARTERLY PROGRESS REPORT

April 1, 1951 to June 30, 1951

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Index No. ORNL-1258
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Classification Changed To:
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By: R. Morrison 7/25/52

Subject Category: Physics and Mathematics

QUARTERLY PROGRESS REPORT

April 1, 1951 to June 30, 1951

March 12, 1952

PROTECTIVE EQUIPMENT EVALUATION PROGRAM
(In Cooperation with the Army Chemical Corps)

E. G. Struxness, Group Leader

[redacted]

OAK RIDGE NATIONAL LABORATORY
Operated by
CARBIDE AND CARBON CHEMICALS COMPANY
A Division of Union Carbide and Carbon Corporation
Oak Ridge, Tennessee

Contract No. W-7405-eng-26

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INTRODUCTION

Recent discussions between representatives of the Army Chemical Corps and the Atomic Energy Commission on matters pertaining to this program led to the following fiscal and technical changes effective with the start of FY 1952:

1. All funds for the support of the program will be provided by the Army Chemical Corps.

2. The scope of the program will be changed from that of developing test techniques to that of studying the penetration through filter media of sub-micron particulates.

The work of the group during this report period has consisted of completing the work associated with the present program, viz., that of adapting standard Chemical Corps tests to radiotracer techniques, and starting work on the proposed program for FY 1952. The LaMer-Sinclair type aerosol generator developments, herein after discussed under Aerosol Generation, have reference to an important requirement in the work to be accomplished next year.

Members of the group have also spent a considerable amount of their time in preparing the following completion reports which are expected to be issued within the next few months:

"Gas Mask Canister Test Against Radioactive Sulfur Dioxide"
"An Evaluation of the Methylene Blue Filter Test"

At the completion of this period, Mr. E. H. Bouton of the Army Chemical Corps' Radiological Division finished his stay of duty at Oak Ridge and returned to the Army Chemical Center for reassignment.

[redacted]

FILTER EVALUATION WITH SOLID AEROSOL (METHYLENE BLUE)

E. H. Bouton, R. L. Quinn, W. D. Cottrell

The methylene blue apparatus was operated during this period for the purpose of determining the efficiency of the thermal precipitator and to check the efficiency of the electrostatic precipitator.

Electrostatic Precipitator Efficiency

The efficiency of the electrostatic precipitator was determined by operating two precipitators in series and by the method of standard and test stains as reported previously[1].

These tests were carried out using S^{35} tagged methylene blue aerosol as a test smoke. In the method of the standard and test stain the activity was counted using an end window G. M. counter and in the case of two precipitators in series the activity was measured using a vibrating reed electrometer.

The results of these tests are given in Table 1.

TABLE 1

EFFICIENCY DATA ON THE ELECTROSTATIC PRECIPITATOR

Test No.	Type of Test	Collection Time (Min)	Penetration %
1	Standard and Test Stain	10	0.0025
2	Standard and Test Stain	10	0.0022
3	Standard and Test Stain (Type 6 Paper)	10	0.0011
4	Two Precipitators in Series	10	0.0013
		AVERAGE	0.0018

1. Protective Equipment Evaluation Program - Quarterly Progress Report, ORNL Y-687, July 1, 1950 to September 30, 1950, pp. 17-19.

In determining the efficiency of the electrostatic precipitator by backing it up with a second precipitator, considerable difficulty was encountered with contamination of the center electrode of the precipitator. It was found that meticulous care had to be exercised in cleaning the center electrode; otherwise, activity was transmitted from the electrode to the cylinder wall and gave penetration values that were too high.

Another necessary precaution was that of minimizing self absorption of the radioactivity. A deposit of methylene blue on the cylinder wall of the precipitator in excess of 0.13 mg/cm^2, obtained during a 10 minute mass concentration run, will result in significant self-absorption. The discrepancy due to self-absorption amounts to approximately 30% of the total radioactivity for a 30 minute mass concentration run.

The average value of 0.0018% for the penetration through the electrostatic precipitator obtained as a result of this work checks the value of 0.0020% reported previously[2].

CWS Type 6 Filter Paper Efficiency

One efficiency determination of CWS Type 6 filter material was made by backing the paper with the electrostatic precipitator and counting the radioactive methylene blue collected by the precipitator.

The value obtained in this manner was 0.0113% penetration.

Thermal Precipitation Efficiency

Methylene blue aerosol was used to test the efficiency of two thermal precipitators; T.P. #I, which had been in use here for some time, and T.P. C-#I, obtained commercially from C.F. Casella Lt'd., London, England.

The efficiency tests were made on a mass basis and were accomplished by passing radioactive methylene blue smoke into the thermal precipitator backed by CWS Type 6 filter paper. The filter paper was counted using an end window G.M. tube and the counts compared with those obtained for the mass concentration of the aerosol.

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2. Ibid. pp. 24-26.

The methylene blue smoke was produced by atomizing a 1% solution of S^{35} tagged methylene blue in the Army Chemical Corps standard methylene blue machine. As in all previous work with methylene blue, the thermal precipitator was mounted directly on top of the filter chamber and the smoke was drawn vertically up into the precipitator. All samples were collected for four minutes at a flow rate of ~6.3 cc/min.

All penetration and background samples were counted for 30 minutes and all mass concentration samples for 10 minutes. The background counts were taken with a clean filter paper in the counter; the paper having first been placed in the filter holder of the methylene blue machine to minimize any errors due to contamination of the paper by the holder.

A total of six background, seven penetration, and five mass concentration samples were taken. These data, together with the calculated average penetration values, are given in Table II.

From inspection of Table II, Column 5, it can be seen that the average mass concentration value of samples 10, 11 and 12 is quite different from that of samples 17 and 18. This being the case, and since some time had elapsed between the taking of the first group and the second, two separate values were used for the mass concentration. The average of samples 11, 12 and 13 was taken for the calculations on precipitator C-#I and the average of samples 17 and 18 for the calculations on precipitator #I. The background values, Table II, Column 3, show no more variation than would be expected from counting alone; hence, the average of all these values was used in making calculations on both precipitators.

The average percent penetration value was calculated according to the following formula:

$$\%P = 100 \frac{(p+b)-b}{(m+b)-b}$$

where (p+b) = penetration + background

(m+b) = mass concentration + background

b = background.

The average mass concentration values were corrected from a 10 minute to a 30 minute basis by multiplying both the 10 minute count and its error by three, since the precision of the count was fixed by the 10 minute count.

TABLE II

EFFICIENCY DATA ON THERMAL PRECIPITATORS C-#I AND #I

Sample No.	Counting Time (Min)	Bkg. C/30 Min	Penetration+ Bkg.C/30 Min	Mass Conc.+ Bkg.C/10 Min	% Penetration	% Efficiency
			Thermal Precipitator C-#I			
1	30	599				
2	30	599				
3	30	579				
4	30		889			
5	30	617				
6	30		771			
7	30	626				
8	30		800			
9	30		1179			
10	10			5230		
11	10			5125		
12	10			5506		
AVERAGE			910 ±153	5287 ± 188	2.03 ±1.01	97.97 ±1.01
			Thermal Precipitator #I			
13	30	581				
14	30		666			
15	30		624			
16	30		688			
17	10			3407		
18	10			4108		
AVERAGE		600 ±16	659 ±319	3758 ±578	0.55 ±0.34	99.45 ±0.34

In evaluating the precision of results, all calculations of errors were made using the 0.1 confidence interval.

The error in the average background count was taken to be the theoretical error of random sampling of a normal population while the errors in the average values of penetration and mass concentration were calculated from the relation:

$$L.E_x = \frac{K}{\sqrt{N}} \sqrt{\frac{N(\Sigma X^2)-(\Sigma X)^2}{N(N-1)}}$$

Where N = number of cases

K = 1.645 for the 0.1 confidence interval.

The error in the average per cent penetration was obtained from:

If $y = f(x, z, w---)$

then $V_y = \left(\frac{\partial y}{\partial x}\right)^2 V_x + \left(\frac{\partial y}{\partial z}\right)^2 V_z + \left(\frac{\partial y}{\partial w}\right)^2 V_w + ---$

In these tests it was assumed that the efficiency of the CWS Type 6 filter paper was the same for both the influent and effluent aerosols. It was further assumed that the same percentage of the activity penetrated into the paper and was not counted in both cases. This would be true only if the particle size distribution were the same in both aerosols.

An insight into the variation in concentration of the smoke produced by this particular machine may be gained by examining the mass concentration values given in Table II. As has been pointed out before, there exists a difference in the two sets of values taken at different times with respect to this period of continuous operation of the methylene blue machine. This difference may be due to a change in the concentration of the methylene blue solution with time. In addition to this, there seems to be a variation in consecutive samples taken one immediately after the other.

Applying the F test of significance

$$F = \frac{S^2}{\sigma^2}$$

where S^2 = observed variance

σ^2 = theoretical variance = count

for samples 11, 12 and 13

$$F = \frac{38,727}{5,287} = 7.3$$

and for samples 17 and 18

$$F = \frac{245,700}{3,758} = 65.3.$$

From appropriate tables we find the maximum permissible values of F to be 2.3 in the first case and 2.7 in the second case. Comparing the observed values of F to their respective maximum permissible values from tables, we find that in both cases the observed values are significantly greater than the maximum permissible values and hence, it must be concluded that the variation in the mass concentration values is greater than would be expected from the variation in the counting rate alone.

Although the cause of these unduly large variations in the concentration of the test smoke was not determined, their effect on the experiment was taken into account when the precision of the results were calculated.

Based on this work it may be stated that we are 90% certain the efficiencies of the precipitators are within the ranges quoted; viz, 99.45 ± 0.34% for precipitator #I and 97.97 ± 1.01% for precipitator C-#I. While both figures are somewhat lower than was expected, it is felt that this does not in any way invalidate the particle size work done previously with these instruments.

It should be pointed out that in these tests the aerosol was drawn vertically up into the precipitators. According to theory, better efficiency should be obtained by drawing the aerosol vertically down into the precipitator against the direction of the convection currents set up by the hot wire of the precipitator. No attempt to verify or measure the magnitude of this effect was made.

AEROSOL GENERATION

E. E. Grassel

Changes

Several changes were made during this quarter on the LaMer-Sinclair type generator being built in this laboratory. These changes can be seen by comparing Figures 1 and 2 in this report with Figures 6 and 8 in the last quarterly progress report[3].

Because of the extreme amount of heat dissipated to the room it was deemed advisable to insulate the box with a 1 inch layer of 85% magnesia covered with a thin layer of asbestos cement. As a result of the extra insulation it was necessary to move the nuclei source to the outside of the box. This allowed the 18-25 watts used by the nuclei source to be dissipated in the room instead of the box, thus preventing the box from overheating.

Stabilizers were added to both the air and vacuum lines to allow a constant and controlled differential in pressure to be applied across the system. The jet in the reheater was enlarged from 1 mm. to 1/4 inch to permit the air through the boiler-nuclei system to be increased to a total of 4 liters/min. A flow meter was added to the diluting system so that the diluting air could be continuously measured. In addition, several small convenience features, such as indicator lights on the intermittent floor heaters and thermocouples at various points in the dry box, were added.

Experimental

With this generator, relatively homogeneous stearic acid smoke in a wide variety of sizes was produced. Smoke made in preliminary runs ranged in particle size from 1.0μ in diameter down to those which showed little or no light scattering when placed in a Tyndall beam of white light. These smaller particles, when grown to five diameters by a technique described by LaMer[4], exhibited some color. The former aerosol was produced by running the nuclei source at 20

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3. Protective Equipment Evaluation Program - Quarterly Progress Report, ORNL Y-768, January 1, 1951 to March 31, 1951, pp. 22,24.
4. LaMer, V. K., Inn, E.C.Y., and Wilson, J. B., J. Colloid Science, 5, 417-96 (1950).

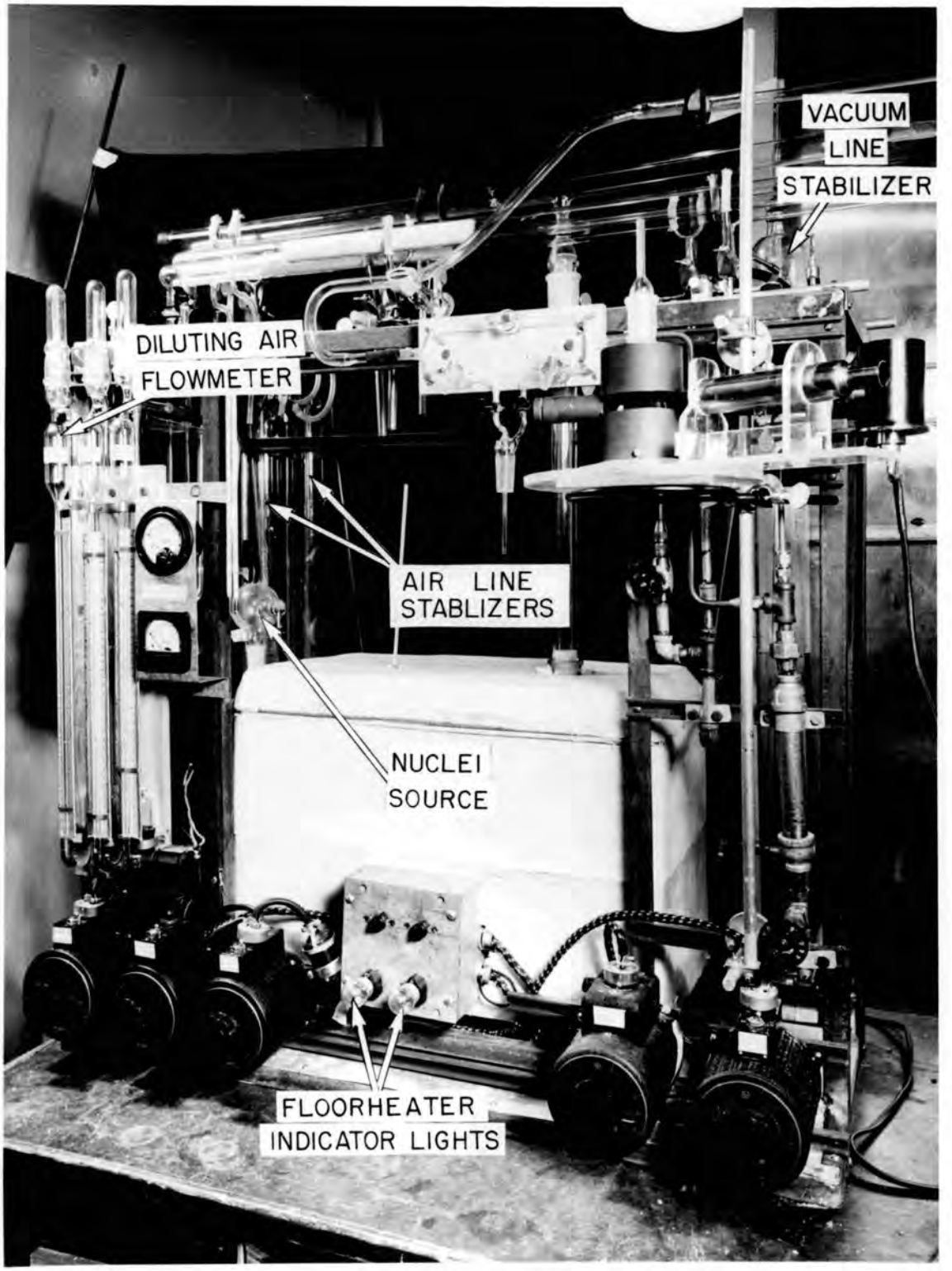

FIGURE I. EXTERIOR VIEW SHOWING ADDITIONS

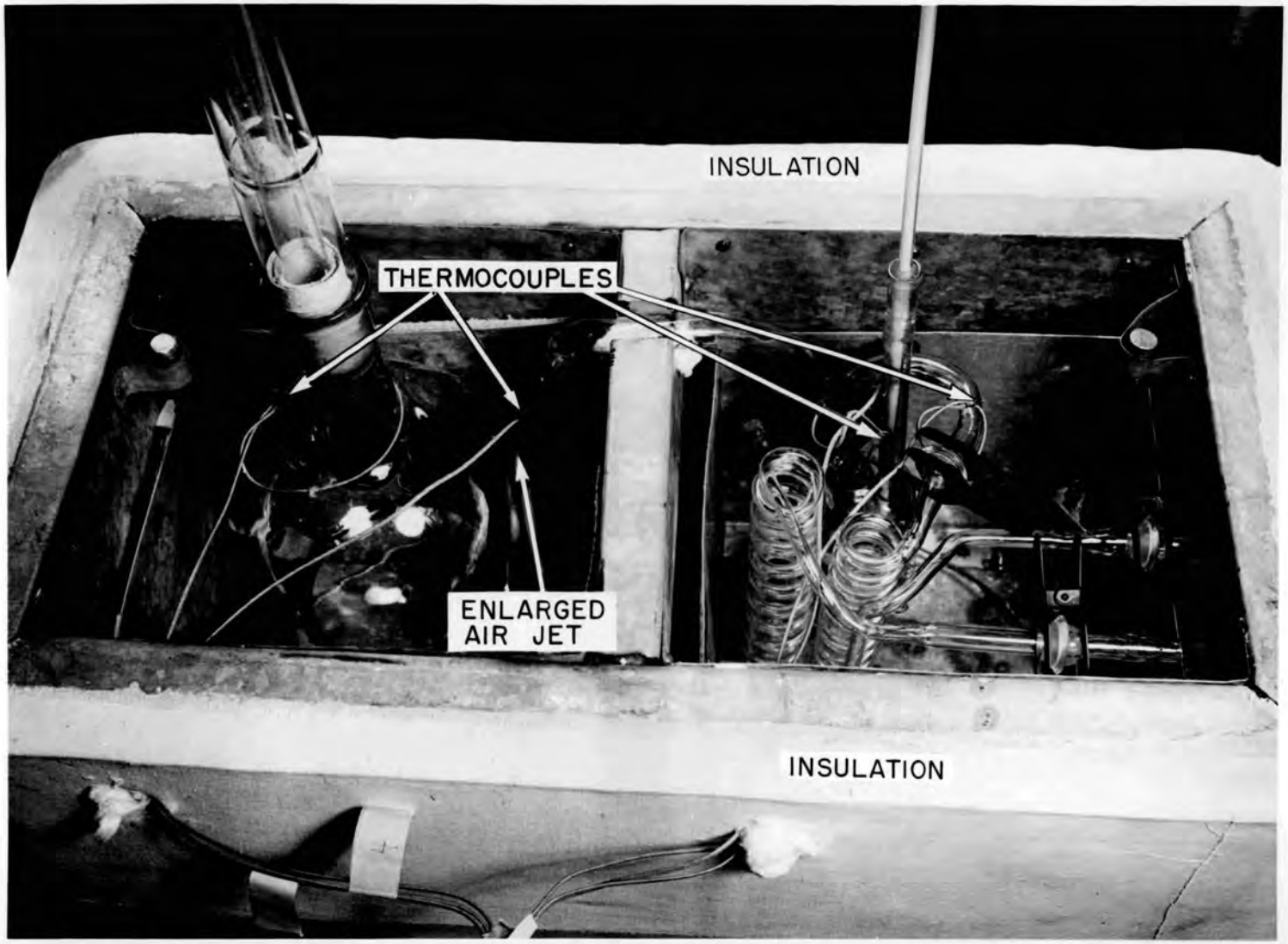

FIGURE 2. INTERIOR VIEW SHOWING ADDITIONAL INSULATION AND THERMOCOUPLES

watts, air through the nuclei source and boiler, each at 1.8 liters/min, boiler temperature at 170°C and diluting the smoke to 15.8 liters/min. Samples of this smoke were drawn over a period of 1/2 hour and the variance in the angular distribution of the reds measured was well within the limits of error of the Owl used. The light scattered was measured from 30° in the forward direction to 150° in the backward direction. The bands fell at approximately 138°, 109°, 61° and slightly less than 30°. This is approximately the proper distribution for the reds in a 0.5μ radius homogeneous aerosol. A sample of the aerosol was collected on Chemical Corps No. 6 paper for a 15 minute interval and the smoke was found to contain about 500 μg of stearic acid per liter of air. This was measured by the difference in paper weights before and after collection. A 30 minute run was also tried but the pressure drop across the paper became excessive causing the flow through the generator to drop, giving meaningless results.

Recommendations

Due to the uneven heat distribution found in this generator box by use of thermocouples, a box of entirely new design and principle of heating is in order. The chimney and diluting mechanism should be run in a constant temperature bath and the diluting mechanism should be redesigned to give a more symmetrical dilution with less turbulence. Also a system of larger diameter tubing, containing fewer constrictions, is needed to convey the sample to where it is to be used or measured. This change in the conveying system is needed because of the extensive deposition of smoke on the walls at bends and constrictions in the small diameter tubing now being used.