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AN IMPROVED REAEROSOLIZATION TEST DEVICE: IT'S DESIGN, OPERATION AND CAPABILITIES

by

Joel M. Klein
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November 1971

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The purpose of this report is to describe the design, operation, and capabilities of an improved laboratory test device that is intended to simulate the reaerosolization of a powder in the field. Its improvement in design over the Monsanto Research Corporation (MRC) reaerosolizer is a result mainly of eliminating the constriction at the top sampling head to permit linear gas flow, using Millipore filter paper over the bottom glass frit for protection against clogging, and changing the Millipore filter paper on the collection head to a fiber mat type paper to prevent loss of powder. The improved apparatus was tested with various CS2, resorcinol, and *para*-aminobenzoic acid powders (particle sizes between 2 and 15 microns MMD) at a range of airflow rates and was found to give a linear relationship between airflow rates and percent reaerosolization. It was also found that varying the pore size (within a certain range) of the Millipore filter paper on the bottom head made little difference in the amounts of powder reaerosolized at various airflows. It is probable that some powder samples can be characterized and rated by their response to different airflow rates. This design is being used locally in lieu of the earlier MRC reaerosolization simulator for the laboratory testing of this powder property.

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1

FOREWORD

The work described in this report was conducted under Project 1W062116A081, Chemical Dissemination/Dispersion Technology (U). This work was started in April 1969 and completed in October 1969. The experimental data are recorded in notebooks 7840, 8027, and 8301.

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DIGEST

The purpose of this report is to describe the design, operation, and capabilities of an improved laboratory test device that is intended to simulate the reaerosolization of a powder in the field. Its improvement in design over the Monsanto Research Corporation (MRC) reaerosolizer is a result mainly of: eliminating the constriction at the top sampling head to permit linear gas flow, using Millipore filter paper over the bottom glass frit for protection against clogging, and changing the Millipore filter paper on the collection head to a fiber mat type paper to prevent loss of powder.

The improved apparatus was tested with various CS2, resorcinol, and *para*-aminobenzoic acid powders (particle sizes between 2 and 15 microns MMD) at a range of airflow rates and was found to give a linear relationship between airflow rates and percent reaerosolization. It was also found that varying the pore size (within a certain range) of the Millipore filter paper on the bottom head made little difference in the amounts of powder reaerosolized at various airflows. It is probable that some powder samples can be characterized and rated by their response to different airflow rates. This design is being used locally in lieu of the earlier MRC reaerosolization simulator for the laboratory testing of this powder property.

CONTENTS

LIST OF TABLES

AN IMPROVED REAEROSOLIZATION TEST DEVICE: ITS DESIGN, OPERATION, AND CAPABILITIES

I. INTRODUCTION.

Controlled field type tests for reaerosolization of CS2 (CS blended with 5% hydrophobic silica) powders have been performed at Edgewood Arsenal.* CS2, a riot control agent, is used to deny terrain to unwanted intruders by reaerosolizing when the intruder enters the contaminated area. Reaerosolization is the process whereby a powder, which has been deposited from a cloud or otherwise on a surface such as the ground, is caused to reform a cloud when disturbed by relatively weak forces.

To study reaerosolization in the laboratory, a suitable device was needed to simulate field testing on a smaller scale. Two techniques have been tried to approximate reaerosolization under controlled laboratory conditions; one using constant vibration as a powder agitator,** and the other using the Monsanto Research Corporation (MRC) glass reaerosolization apparatus.† In the absence of field tests of powder reaerosolization, it is not possible to correlate these laboratory techniques directly with reaerosolization in the field. Of these two techniques, the MRC device offered the better means of testing a powder for apparent reaerosolizability because the results obtained with it were more reproducible and the gas flow and humidity could be controlled.

The MRC apparatus consists of a Pyrex glass tube 2-7/8 inches in diameter and 14 inches long with a coarse glass frit, used as a gas diffuser, located near the bottom. The tube is provided with a side arm, located 5 inches above the frit, through which a powder sample is introduced. A small quantity of nitrogen flowing through the frit distributes the powder sample particles over the glass frit, after which a gas flow of 200 cc/sec is directed through the powder bed distributed on the frit. The reaerosolized portions of the sample are carried up the tube by the gas flow and collected on a Millipore filter at the upper portion of the glass apparatus. This filter is removed and weighed to determine the quantity reaerosolized.

Several disadvantages result from the design of the MRC reaerosolizer. Because the powder initially is deposited directly on the glass frit, the frit becomes clogged with powder, which changes its porosity. As a result, the airflow through the frit is altered, and the reproducibility of the results is affected. The tapering of the column at the upper (sampling) head also alters the airflow pattern. This introduces a changing stream velocity profile and creates turbulence at the sampling head. Furthermore, the sampling filter used is a membrane type filter (Millipore), which has a tendency to lose powder when the sampling head is removed. If the sample size is kept small, the membrane filter usually is satisfactory, but this restriction on size is undesirable.

A test device built at Edgewood Arsenal, having several distinct design advantages over the MRC device in characterizing the apparent reaerosolization of powders, is described in this report.

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*MacLeish, Joseph H., and Frickel, Robert H. EATR 4302. Portable Chamber Field Test of CS2. June 1969. UNCLASSIFIED Report.

**Klein, J. M. EATM 142-4. Reaerosolization of CS2 for Terrain Denial. May 1969. UNCLASSIFIED Report.

†Headley, W. H., Feairheller, W. R., Richardson, G. A., and Zanders, D. L. Monsanto Research Corporation. Quarterly Progress Report No. 2. Contract DAAA15-68-C-0006. Studies of the Surface Chemistry of Solids in Dissemination. January 1968. UNCLASSIFIED Report.

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II. EXPERIMENTATION.

Three modifications have been made in the Edgewood Arsenal test device to correct the defects found in the MRC reaerosolizer. The fritted glass diffuser is protected by a Millipore filter from clogging by the powder. The glass column is now straight from the diffuser filter to the sampling filter. Finally, a depth filter (type 1106B) is used in place of the Millipore filter at the sampling heads to better retain the collected powder.

A. Materials.

1. Reaerosolization column—24 inches long, 80 mm in diameter, flanged ends, attached side tube 25 mm in diameter with 24/25 ST ground glass joint.

2. 24/25 ST Glass stopper.

3. Collection head.

4. Reaerosolization head.

5. Two O-ring seals—90 mm in diameter, Labglass, Vineland, New Jersey.

6. 1106B Filter paper—produced by Hurlbut Corporation, South Lee, Maine.

7. Millipore filter paper—pore sizes 0.45 and 1.2 microns.

8. Two McCarter's clamps—Labglass, Vineland, New Jersey.

9. Airflow gage-rotameter—registering up to 9125 cc/min.

10. Vacuum source.

11. Sample injection tube—handmade.

12. 0.1% Aerosol O.T. solution.

13. Treated resorcinol—resorcinol preground in a jet mill to less than 10 microns diameter and blended with 5% hexamethyldisilazane-(HMDS) treated Cab-O-Sil HS-5.

14. Treated *p*-aminobenzoic acid (PABA)—PABA preground in a jet mill to less than 10 microns diameter and blended with 3% hexamethyldisilazane-treated Cab-O-Sil HS-5.

15. Treated *o*-chlorobenzalmalononitrile (CS)—CS preground in a jet mill to less than 10 microns diameter and blended with 5% Dow Corning silica X95527.

B. Methods.

The operating procedure used with this new apparatus is given in the appendix. A schematic diagram is shown in figure 1.

In order to evaluate this instrument, a series of experiments was conducted to investigate the effects of the following variables on the reaerosolization of powders:

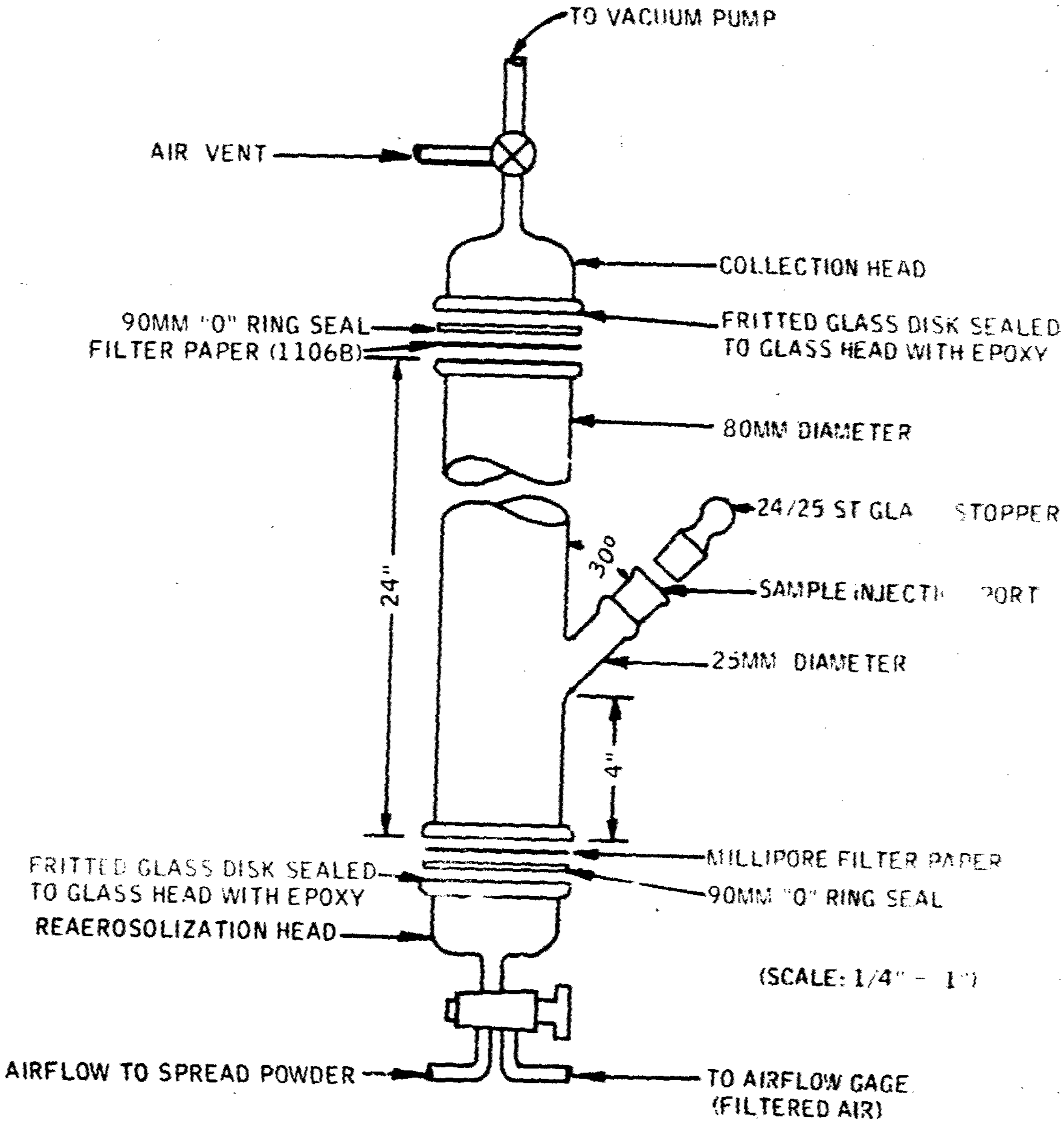

Figure 1. Reaerosolization Test Device

1. Instrumental variables
   a. Airflow rate
   b. Pore size of the filter-air diffuser
2. Sample variables
   a. Quantity of powder

b. Type of powder

c. Density of powder

3. Other variables

a. Sequential sampling (see below)

b. Wall loss

Where possible, only one variable was changed in any experiment. The quantity of material obviously could not be reproduced exactly in each run. Each experiment was run at least twice.

In order to determine when the major portion of the sample impacted on the sampling filter and whether this depended on the nature of the sample, a series of experiments was conducted in which the sampling head was disconnected quickly from the column during the run, the filter removed and replaced with a new tared filter, and the sampling head reattached to the column. While the filter was being changed, there was no airflow in the column, and thus, no material was lost. In this fashion, it was possible to follow the rate of the reaerosolization. After each run, the powder remaining on the diffusing filter was weighed, and the amount of powder adhering to the walls was determined from the difference.

III. RESULTS.

A. Instrumental Variables.

The results of these experiments are summarized in tables I and II. The dependence of reaerosolization on the airflow rate is linear, as shown in figures 2, 3, and 4. These experiments show that the linear dependence of reaerosolization is independent of material type.

To verify that the observed linearity is not accidental, the regression coefficient, r, was calculated for each result using the equation:

$$r = \frac{n\sum_{i=1}^{n} x_i y_i - \left(\sum_{i=1}^{n} x_i\right)\left(\sum_{i=1}^{n} y_i\right)}{\sqrt{\left[n\sum_{i=1}^{n} x_i^2 - \left(\sum_{i=1}^{n} x_i\right)^2\right]\left[n\sum_{i=1}^{n} y_i^2 - \left(\sum_{i=1}^{n} y_i\right)^2\right]}}$$

Table I. Reaerosolization Data in Filter Paper Variation Study

In tables I and II, the sample is 95% resorcinol and 5% Cab-O-Sil ST-1.

Filter pore size	Percent reaerosolized (by weight)			
	Airflow (cc/min)			
	4850	5900	6950	8050
microns	%			
1.2	17.87	22.90	41.14	52.97
0.45	12.27	20.05	32.83	60.64

Table II. Reaerosolization Data in Sample Weight Variation Study

Approx sample wt	Percent reaerosolized (by weight)					Regression coeff
	Airflow (cc/min)					
	3850	4850	5900	6950	8050	
gm			%			
0.1	2.74	18.75	16.13	38.66	41.77	0.95
0.3	4.51	17.87	22.90	41.14	52.97	0.99
0.5	3.50	12.70	23.34	36.27	49.34	0.99

where

X_i = the airflow rate of the particular experiment

Y_i = the reaerosolization result in percent

n = the total number of points*

The closer the value of r is to 1.00, the greater the probability that the data are indeed linear. The values of the regression coefficient, listed in table II, are all greater than 0.95. This shows that the results are indeed linear at the 99.5% confidence interval.

To test the effect of the air diffusion pattern on reaerosolization, powders were reaerosolized using Millipore filters with different pore sizes as diffusers. The construction of the Millipore filter is such that, for filters of different pore size, the total open surface area is approximately the same. Thus, for the filters used in these experiments--pore sizes ranging from 0.45 to 1.2 microns--the flow rate of air or gas through the filter would be about the same under similar operating conditions. The technical data for these filters provided by the Millipore Corporation show airflow rates varying between 10 and 15 liters of air per minute per square centimeter of filter surface.** The air diffusion patterns, however, are different for the different filters as the size and number of pores on the surface are different. The results of these tests show that there is no significant variation in reaerosolizability within the range of pore sizes used.

B. Sample Variables.

Tables II and III present the results of experiments conducted to show the effect of varying sample size and sample type. These results are plotted in figures 3 and 4. As indicated, we can state with considerable certainty that in all cases, the dependence of reaerosolization on airflow rate was linear. The regression coefficients confirm this observation.

In table IV the results are presented from experiments conducted with resorcinol powders that had been blended with HMDS-treated Cab-O-Sil. Each of these powders was prepared from the

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*The general equation for the regression coefficient can be found in most standard statistics texts. The form of the equation for a linear regression coefficient used here is the equation used in computer programs.

**Millipore Corporation, Bedford, Massachusetts. Catalog mc/1. 1970.

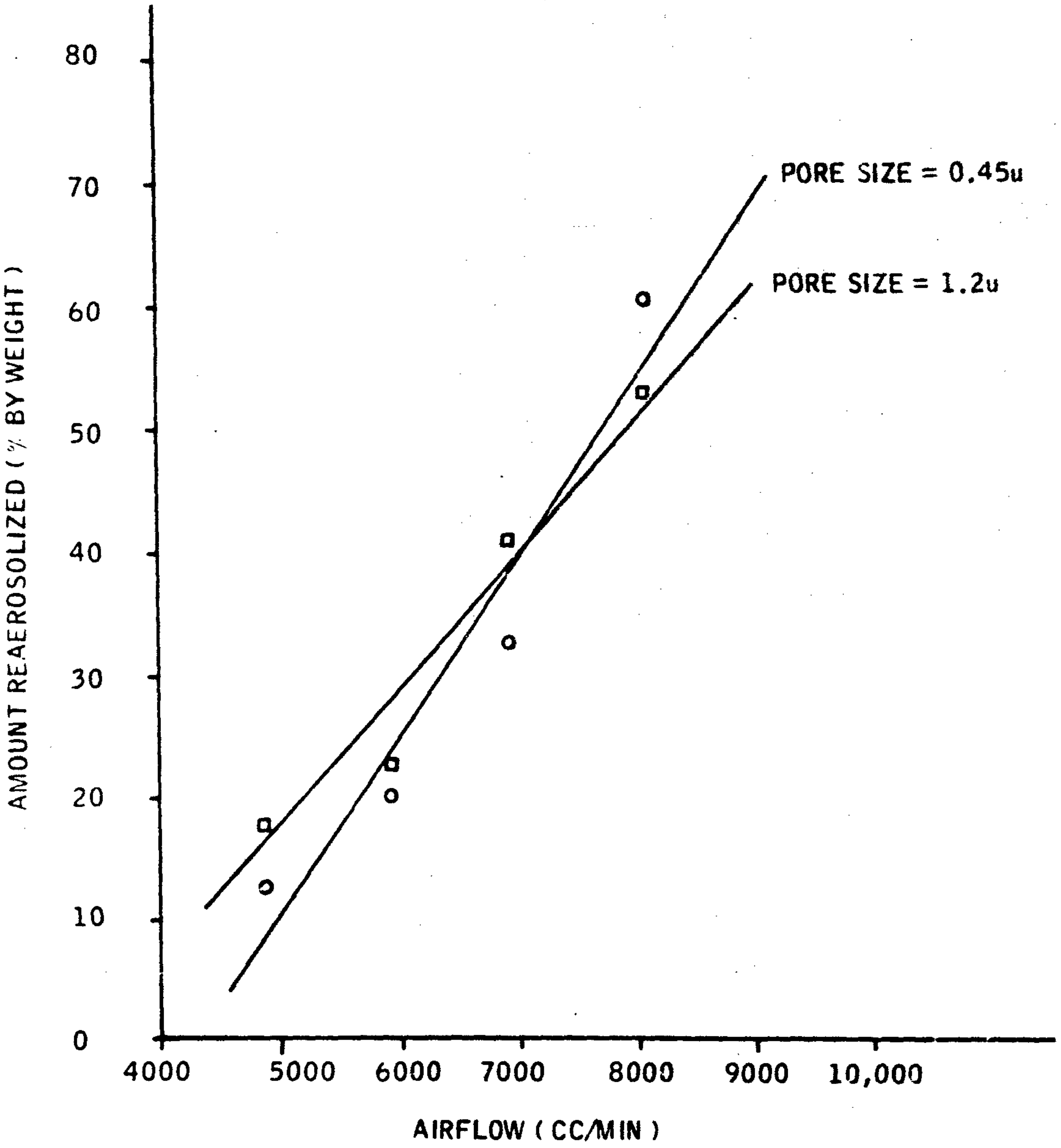

Figure 2. Millipore Filter Paper Variation Study

same lot of preground resorcinol powder, and each had the same particle size distribution. The only major physical difference between the powders was the bulk density. These results show that the dependence of reaerosolizability on density is not established, but that a weak correlation between these parameters may exist.

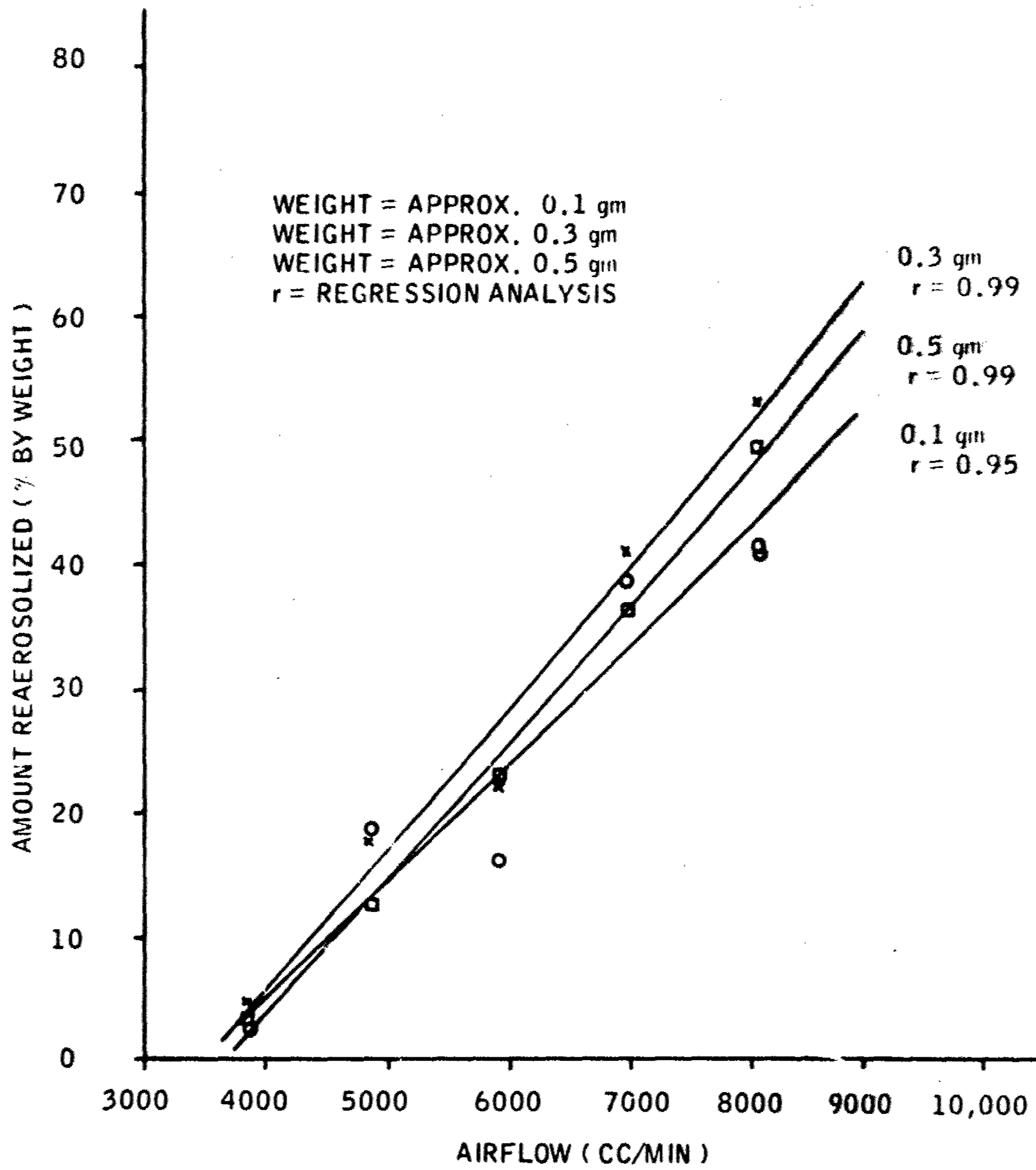

Figure 3. Sample Weight Variation Study

C. Other Variables.

The results of typical sequential sampling experiments are presented in table V. Numerous experiments were conducted, but only selected results are presented here. The amount of material adhering to the walls of the reaerosolizer was calculated by totaling the amount recovered on the top filter with the amount remaining on the bottom filter. The balance of the material was assumed to be adhering to the wall. The wall loss typically was 20%.

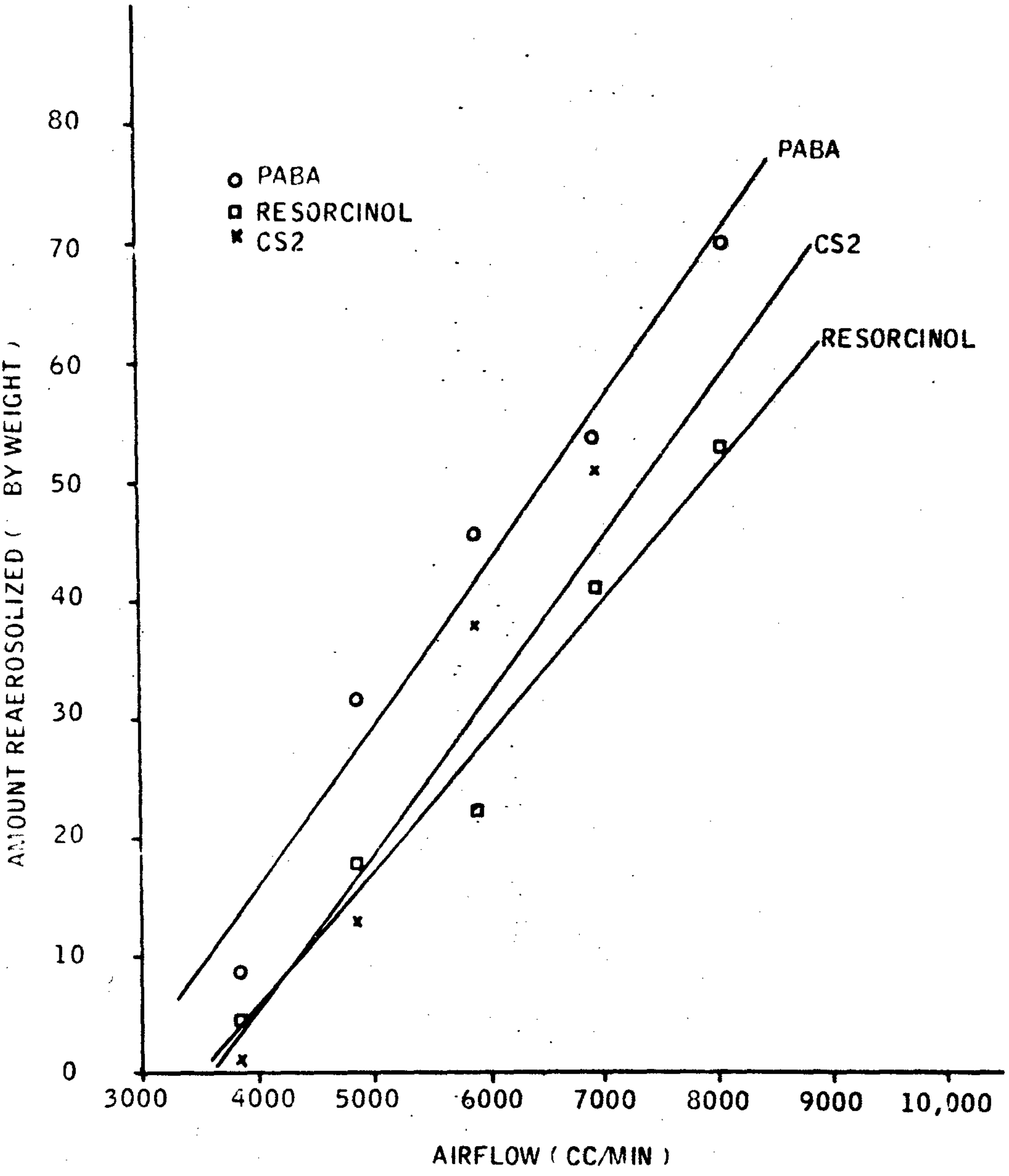

Figure 4. Sample Type Variation Study

IV. DISCUSSION.

It can be seen in figure 3 that a variation in sample size with different airflows yields a linear correlation for each sample weight, and the plots differ very little from each other. This is somewhat surprising in that the sample consists of a nonlinear particle size distribution; thus, the weight of the particles leaving the bed would be expected to change nonlinearly at different airflow rates. (It is conceivable that the air velocity through the pores of the Millipore filter has enough

Table III. Reaerosolization Data in Sample Type Variation Study

Treated sample	Percent reaerosolized (by weight)					Regression coeff
	Airflow (cc/min)					
	3850	4850	5900	6950	8050	
	%					
Resorcinol	4.51	17.87	22.90	41.14	52.97	0.99
p-Aminobenzoic acid	8.87	31.72	45.76	53.82	70.16	0.98
CS2	1.31	13.04	38.00	51.19	52.71	0.96

force to lift all particles in the 1- to 30-micron size range from the surface of the filter at the time airflow starts.) Also, a variation in sample weight (0.1 to 0.5 gram) seems to have little, if any, effect upon the percentage of powder reaerosolized.

The quantity of material used in each run corresponds to a contamination density of 20 to 100 gm/sq m. This brackets the currently recommended density of 50 gm CS2/sq m for terrain denial.* These results indicate that this variation in the quantity of material used will not affect the performance of the agent in the laboratory test.

The use of different pore sizes of Millipore filters on the lower head resulted in little difference between the amounts of powder reaerosolized at various airflows (figure 2).

It is probable that some samples can be differentiated from each other by data plots showing their relationship of percent reaerosolization and airflow (figure 4). Furthermore, it may be possible to differentiate between samples having the same base material but containing different silica substrates. Further work is in process to investigate this possibility.

Laboratory use of this improved reaerosolization test apparatus has shown it to be of significant value in powder research, and that it is a more practical design than the MRC unit. Maintenance and selection of the glass frit used at the point of reaerosolization had been critical. This critical point has been eliminated in this new design because even a change in filter pore size does not qualify or change the reaerosolization results noticeably.

This new test device meets the requirements for the evaluation of powders in the laboratory. Field tests of powder reaerosolization run in conjunction with this test device are needed to show whether it does indeed simulate reaerosolization. Reaerosolization can be measured in the field by comparing the quantity of material kicked up forming an effective aerosol to the amount of material remaining on the ground. A carefully controlled field test device is also needed to kick up powder from the ground in a reproducible manner.

V. CONCLUSION.

A new laboratory test device has been fabricated and has demonstrated several advantages over the MRC reaerosolization simulator. This device is being used locally in lieu of the earlier MRC apparatus as part of the powder characterization program.

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*Klein, *op. cit.*

Table IV. The Effect of Density on Reaerosolizability

All samples consist of 95% by weight resorcinol powder, preground to 5-micron MMD, and 5% HMDS-treated Cab-O-Sil.

Sample preparation	Density	Reaerosolization	
		4850 cc/min	8050 cc/min
	gm/cc	%	
Plastic 'V' blender	0.34	12.27	43.36
Steel 'V' blender	0.35	10.66	41.07
'V' blender with bar	0.64	18.56	60.22
Drum blender	0.42	8.18	44.32
Drum blender with rubber stoppers	0.40	13.84	52.76
Drum blender with ceramic rods	0.37	11.04	47.97

Table V. Sequential Reaerosolization

All runs were conducted with resorcinol powder drum-blended with 5% HMDS-treated Cab-O-Sil, run at an airflow of 4850 cc/min.

Run No.	Reaerosolization					
	At time (min)				On bed	On wall
	10	20	30	40		
	%					
1	11.38	9.08	8.96	5.70	17.33	47.55
2	24.96	6.01	5.40		44.99	18.42
3	17.81	18.77	10.73	6.30	33.07	14.00
4	15.40	14.19	11.03		33.98	25.40
5	21.82	16.24	11.16		32.73	18.05
6	22.61	13.56	13.06		34.44	9.83

APPENDIX

OPERATION OF THE MODIFIED REAEROSOLIZER

The column first is cleaned with Sparkleen and water, rinsed well with water, and given a final rinse with a 0.1% solution of Aerosol O.T. After the column is dried in a stream of dry air, a piece of Millipore filter paper is placed on the bottom head, which is clamped to the main column with a McCarter's clamp. The airflow gage—a rotameter—is connected to the bottom head with Tygon tubing. A piece of weighed 1106B filter paper is placed on the top or sampling head, and this filter paper in turn is clamped to the column with another McCarter's clamp. The vertical tube on the top head is connected to the vacuum pump by Tygon tubing. A powder sample of about 0.3 gram is weighed and placed on the Millipore filter paper by inserting the sample injection tube in a port in the side of the column. The glass stopper is inserted to close the port. The valves on the top and bottom heads are turned to open the system from the airflow gage to the vacuum pump. Then a small quantity of dry air is carefully introduced through the bottom head to spread the powder evenly. Now, with the top head valve closed, the vacuum pump is turned on, and the top head valve is carefully opened to the preselected airflow. Upon reaching the desired airflow, a stop watch is started. At the end of 10 minutes, the top head valve is turned so that the air vent tube leaks air into the system. The McCarter's clamp at the top head and the glass stopper are now removed. With a finger placed over the air vent tube, the resulting vacuum holds the powder to the filter paper. The head is gently lifted from the column and inverted. The finger is now removed from the air vent and the vacuum turned off. The powder and filter paper are weighed to find the percent of powder reaerosolized.