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MRC PROJECT NO. 4530

STUDIES OF SURFACE CHEMISTRY OF SOLIDS IN DISSEMINATION

REPORT NUMBER 3

Third Quarterly Progress Report

February 1968 – April 1968

by

W. H. Hedley
W. R. Feairheller
R. L. Long
R. A. Vacchiano

June 1968

DEPARTMENT OF THE ARMY
EDGEWOOD ARSENAL
Research Laboratories
Physical Research Laboratory
Edgewood Arsenal, Maryland 21010

Contract DAAA-15-68-C-0006

MONSANTO RESEARCH CORPORATION
DAYTON LABORATORY
DAYTON, OHIO 45407

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FOREWORD

The work described herein was authorized under Project 1B522301A081, Dissemination Research on Chemical Agents (U). The work was started in February 1968 and completed in April 1968.

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ACKNOWLEDGMENTS

This program is being monitored by the Edgewood Arsenal Physical Research Laboratory, Research Laboratories, Edgewood Arsenal, Maryland, with Mr. James D. Wilcox serving as Contract Project Officer.

Others contributing efforts on this contract at Monsanto Research Corporation include Mr. George A. Richardson and Mr. James Baker.

DIGEST

The objectives of this program are (1) to develop a quality control test for CS-2 which will insure that the product accepted will be reaerosolizable, and (2) to study the surface chemistry of powders to determine the factors that limit their dissemination by pneumatic and explosive means.

A performance index is suggested that should be a measure of how well a powder performs in terrain denial applications, and an experimental program has been designed to determine quantitatively the relative value of several powders for this application. The suggested approach should make it possible to determine the optimum values of reaerosolizability, hydrophobicity, and particle size for terrain denial powders.

New coatings have been formulated that should maximize certain effects such as hydrogen bonding, hydrophobicity, antistatic qualities, and lubricity, and these coatings have been applied to portions of a single sample of Cab-o-sil in preparation for mixing with CS and testing for disseminability.

Presized samples of CS, cornstarch, glycine, resorcinol, and saccharin were coated with Cab-o-sil and HMDS-coated Cab-o-sil from a single batch of silica powder by blending in an Oster blender. The disseminability of these powders differed significantly, depending upon which substrate particles were used and whether the Cab-o-sil was HMDS-coated or not.

Tests on CS-2 powders showed that reaerosolizability in all cases increased with increasing sample size. A sharp rise in reaerosolizability was observed for some powders at a sample thickness of 0.04 cm, but this thickness is greater than would be practical for use under field conditions. Powders that had been exposed to humidity were considerably less reaerosolizable than those that were stored dry. Multiple correlation analysis of previous data shows a good correlation between the logarithm of reaerosolizability and the reciprocal of percent water, hydrophobicity, the reciprocal of fluid density, and the ratio of hydrophobicity to fluid density. It appears likely that humidity, therefore, will have a considerable effect upon the performance index of terrain denial powders.

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STUDIES OF SURFACE CHEMISTRY OF SOLIDS IN DISSEMINATION

1. INTRODUCTION

This contract involves two related but individual studies: (1) investigation of the surface chemistry of coated solids and the effects of these coatings on disseminability by explosive and pneumatic means, and (2) development of quality control tests that characterize CS-2. Although information from each of these studies may influence the other, the two objectives are distinct and separate.

The investigation of the surface chemistry of powders in Task I relative to their pneumatic and explosive dissemination is needed because previous research has yielded results that are as yet unexplainable. Highly flowable powders, such as CS-2, which are more readily disseminable pneumatically than other types of CS, are little better than the other types when disseminated explosively. Also, if CS-2 disseminates pneumatically better than other types of CS, one wonders if there are other types of coatings that would allow treated powders to be disseminated still more effectively than CS-2. The objectives of Task I are therefore to apply different types of coatings to powders, characterize them, and study their performance in dissemination tests in order to gain insight into the mechanisms that limit dissemination of powders.

The goal of Task II is the development of simple, but significant, quality control tests to aid in the selection of the best large scale coating process for the product and will be the basis for preparation of specifications on the mass-produced material. When producing materials on a large scale, controlling all processing variables as closely as possible in small scale laboratory preparations is difficult. Changes occur in the process that may affect the quality of the product result. Quality control tests must be developed to differentiate between surface-treated CS-2 that will perform effectively and that which will not be so effective. Until the variables affecting the performance of CS-2 are identified and simple tests devised to measure these variables, the optimum process to be used in producing CS-2 on a large scale cannot be selected.

2. ANALYSIS OF THE PROBLEM

2.1 What is the Problem?

The biggest problem in the current investigation of use of CS-2 for terrain denial applications is to define which powders perform best in actual usage. Once we know this, we can perform many tests and analyses on these powders (assuming that truly representative samples are used) and ultimately find quality control tests which differentiate between good and bad samples. Our most pressing need, therefore, is to experimentally test powders in some meaningful way so as to obtain samples of powders we can confidently and quantitatively classify according to usefulness.

2.2 Major Factors Involved

At least three major properties affect the problem of maximizing the effectiveness of CS-2 for terrain denial applications: (1) reaerosolizability, (2) persistence, and (3) logistic practicality (Ref. 1). The thickness of spreading is important because at the concentration of 50 grams per square meter that has been experimented with, the powder requirement would be quite high (143 tons per square mile). Field tests have definitely established that CS-2 is sufficiently reaerosolizable to be definitely effective in terrain denial. Field tests have also indicated that CS-2 is persistent when deposited on the ground, even if it comes in contact with moisture.

Certain properties that are desirable for CS-2 are obvious. One is persistence for a long period of time. Another is its effectiveness when distributed in small concentrations per square foot. The effectiveness of a powder in denying a square foot of terrain will probably be a function both of its ability to persist (neither blow away excessively nor degrade quickly upon weathering) and upon the concentration at which it is deposited.

The magnitude of other properties to be desired for terrain denial powders are not obvious. A reaerosolizability of at least the minimum amount in order to be effective is a prerequisite. This minimum level has not yet been established. Maximum reaerosolizability for a powder might be a disadvantage because it could conceivably lead to a lower persistence and yet not be any more effective in denying terrain. Even the desired level of so basic a property as particle size has not yet been established. Since CS type powders do not need to penetrate the respiratory system deeply in order to be effective, one could seriously consider using particles larger than the usual inhalable range. It is possible that they would be adequately reaerosolizable and yet more persistent. Hydrophobicity might also have an optimum value since powders that tend to completely reject water might have more electrostatic agglomeration effects, possibly adversely affecting their reaerosolizability.

2.3 Desired Goals

Upon reflection, it seems reasonable as an overall goal that the best powder would be one that would deny an area of ground for the longest time per gram of material deposited. Such a powder would be relatively effective, long lasting, and logistically practical. These again are the qualities mentioned above as being desirable. This expression can be quantified in the form of a performance index (Equation 1).

$$P.I. = \frac{T_d A}{w} \qquad (1)$$

P.I. = performance index

T_d = length of time the area is denied effectively, days

A = area denied, meters^2

w = weight of agent required, grams

If a concentration of 50 grams of powder per square meter would effectively deny this terrain for 60 days, the performance index would be 1.2 day-meter^2 per gram. This index obviously rates more highly the powders that have the optimum combination of two of the major properties of interest, persistence and logistic practicality.

This performance index also indirectly includes the effect of other properties that have been mentioned as being possibly important. It includes the effect of optimum reaerosolizability since values of reaerosolizability that are either too low or too high would adversely affect T_d. It also includes the effect of optimum particle size since this may also affect T_d. The optimum hydrophobicity, whether high to minimize the caking effects of moisture or low to decrease electrostatic effects, would also influence T_d.

2.4 Implications of the Performance Index Concept

The next step is to determine performance index on a number of powders that have weathered in an identical manner. This can be done by spreading test plots of these powders at various concentrations on the ground at Edgewood Arsenal exposing them all to uniform weathering and running realistic reaerosolizability tests on them as a function of time. Two approaches to making realistic reaerosolizability tests have been described recently (Ref. 2, 3). In one, a one-foot diameter wooden disk is dropped inside a man-sized enclosure onto a plot of powder that has been freshly laid on the bottom, and samples are taken at various heights as a function of time. In the other, a larger, longer enclosure covering a plot of powder through which people can walk is equipped with sampling devices. The virtue of these tests is that samples of powder are used that are potentially small enough to be accurately characterized, yet the testing is done in such a way as to avoid the effects of meteorological variables. These tests should be done on field-weathered samples that have been statistically designed to include powders with different hydrophobicities, particle size, reaerosolizability, and bulk density since there is reason to believe that these properties are important. The effect is an important factor in the performance index.

Another important step is the development of laboratory tests that predict the results found on these larger scale tests since the larger scale tests, though necessary, will be too cumbersome for use in quality control. These tests must be performed on the samples of powder used in the larger scale testing from which representative portions have been set aside for lab scale testing. We would then have samples that can be characterized and for which we have a basis for classifying as good or bad. The lab scale tests will probably have to measure reaerosolizability either directly or indirectly, but unlike the current tests they will probably have to be done on artificially "weathered" samples.

The mention of artificially "weathered" samples brings up another subject which will require investigation. Along with the field reaerosolizability tests suggested above, we should also examine the field plots to determine the mechanism whereby the powder becomes ineffective (whether by being blown away, caked together, etc.). These studies would then act as a guide for formulating laboratory samples of "weathered" powder.

Completion of the studies mentioned above should provide a firm basis for formulating quality control tests for CS-2 terrain denial powders. Other studies will then become pertinent, however. Studies of other combinations of hydrophobicity, particle size, and density not initially tried may be indicated. Different percentages of Cab-o-sil, different amounts of hexamethyldisilazane (HMDS), different production methods, etc., then become extremely important. Also, the possibilities of other coating materials to augment or supplant HMDS can be investigated, once a mechanism for establishing relative merit of powders has been established.

We believe that active pursuit of the terrain denial program should be organized along the lines indicated above. It can provide a clear goal upon which the various groups working on this problem can focus and thus provide a unity to the several efforts on this problem, a coordination which will be necessary (Ref. 4) if this problem is to be solved quickly.

3. TASK I - NEW COATINGS-FORMULATION APPLICATION AND EVALUATION

The purpose of work described in this section was to study the surface chemistry of powders so as to develop an understanding of the variables that determine their disseminability. Two approaches are being currently pursued: in the first, new coatings that are designed to maximize certain properties of powders (such as hydrogen bonding, hydrophobicity, etc.) are to be applied to Cab-o-sil in place of hexamethyldisilazane (HMDS), and these powders are then to be used to coat the particles of one substrate powder and will be studied to see how they affect disseminability. In the second approach, Cab-o-sil and HMDS-treated Cab-o-sil from the same batch are used to treat different substrate powders, which are then studied to determine if the substrate powder affects the performance after coating.

3.1 Effect of New Coatings on Cab-o-sil Disseminability

In this study, particles of one substrate powder from a single homogenized batch are to be coated with Cab-o-sil particles (also from a single homogenized batch) which have in turn been coated with new coating materials in place of HMDS. The initial coating materials chosen have been selected to maximize a certain property (such as hydrophobicity or hydrogen bonding) in order to isolate and enhance the surface forces suspected of playing a major role during dissemination (Ref. 7). These coatings include (1) pentaerythritol tetrakis(trifluoroacetate) referred to as PTFA, a compound capable of forming strong hydrogen bonds to a Cab-o-sil surface; (2) hexafluoroacetyl acetone, referred to as HFAA, a compound capable of reacting with surface silanol groups; (3) OS-45, a commercial silicate fluid (Monsanto Co.); and (4) trioctylpropyl ammonium bromide, an antistatic agent.

Since these coatings will eventually be compared with the CS type 2 coating, the following rationale was developed to facilitate this comparison. Cab-o-sil HS-5 has been reported to possess a surface silanol concentration of three groups/$(m\mu)^2$ and a surface area of 270 m^2/g. When coated with hexamethyldisilazane (HMDS) each mole of HMDS reacts with two SiOH groups. The number of SiOH groups per gram of Cab-o-sil is then:

$$\left(\frac{3\ \text{groups}}{(m\mu)^2}\right)\left(\frac{10^9\ m\mu}{m}\right)\left(\frac{270 m^2}{g}\right) = 8.1 \times 10^{20}\ \text{groups/g}$$

Since 1 mole of HMDS =161 g, the theoretical quantity of HMD to react with all available SiOH groups is:

$$\frac{\text{g HMD for theoretical coverage}}{\text{g of Cab-o-sil}} =$$

$$\frac{8.1 \times 10^{20} \text{ molecules}}{6 \times 10^{23} \text{ molecules/mole}} \cdot \frac{161 \text{ g/mole}}{2} =$$

$$0.108 \text{ g} = 0.67 \text{ mmole}$$

In practice, 40% of the treated Cab-o-sil weight is HMDS; thus 0.67 g of HMDS is used. This represents a sixfold (0.67/0.11) excess over that required theoretically. Hence, 4.0 mmoles of HMD are used for each g of Cab-o-sil (or 0.07 mole for the 18-gram samples of Cab-o-sil used.

Since each mole of pentaerythritol tetrakis(trifluoroacetate) theoretically covers four SiOH groups, 2 mmoles were used for each gram of Cab-o-sil (approximately 50% by weight). Each mole of hexafluoroacetyl acetone theoretically covers 2 SiOH groups, hence, 4 mmoles were used for each gram of Cab-o-sil (approximately 47% by weight). Since OS-45 is a proprietary material whose exact composition has not been revealed, we used the same quantity as that for HMDS, 40% by weight. The trioctylpropyl ammonium bromide is on order thus, no coatings were made with this compound.

In the experimental preparation of coated Cab-o-sil 16.0 grams of HFAA was placed in an Osterizer blender and 18.0 grams of Cab-o-sil HS-5 was added slowly. When the addition was completed, the mixture was blended for two minutes. Only the lowest speed was used. This material was placed in a jar for future use.

Since PTFA is a solid, it was necessary to dissolve it in a solvent and apply it as a solution in order to spread it evenly over the Cab-o-sil surface. Twenty grams of PTFA were dissolved in 500 ml of $CHCL_3$ and a portion of this was placed in an Oster blender. The Cab-o-sil was then added slowly until the blender began to vibrate. More of the PFTA solution was then added. When all the Cab-o-sil had been added, the mixture was blended at low speed for two minutes. The mixture was then placed in an oven to dry at 60°C for two hours. This drying process was too slow, so the mixture was spread in a thin layer on aluminum foil and allowed to dry overnight. The dried powder formed a cake, which was broken up using an Osterizer blender for 15-30 seconds. The resulting powder consisted of large agglomerates so the container was rolled overnight at 288 RPM in an attempt to break them up. This procedure did not break up the powder sufficiently so it was ball milled for 4 hours with Burundum cylinders. This reduced the particle size considerably; however, particle size counts have not yet been obtained.

The same process was used for 12 g of OS-45 except that heptane was used as the solvent. The powder produced was also badly agglomerated. The same procedures were used to break up the millimeter-size agglomerates, and it was again found that ball milling was necessary in order to effectively reduce their size.

3.2 Studies of the Effect of Substrate Powders on Disseminability

Since large differences in the behavior of samples of CS-1 and CS-2 have been noted, the question arises as to whether the differences are due to variations in the Cab-o-sil coating, variations in the CS particles that are coated, or whether both effects are important. In order to shed light on this question, we decided to take one specific batch of Cab-o-sil, coat different particles with it, and test the resulting mixture to see if the variation in substrate powders significantly affected the disseminability of the mixture. Having removed such variables as specific lot of Cab-o-sil particles, size of Cab-o-sil particles, specific surface area of Cab-o-sil, and nature of chemical surface on the Cab-o-sil particles, we should have been in the position to attribute differences in the disseminability to characteristics of the coated powder particles.

Powders selected for this study included CS, cornstarch, glycine, resorcinol, and saccharin. These powders were selected since characterized samples of each of them were available, and these samples are all known to contain a large fraction of particles with sizes of the inhalable range. A sizable quantity of Cab-o-sil HS-5 was blended and a sample was withdrawn. Eighteen grams of this Cab-o-sil was poured into an Oster (Model 641) blender into which twelve grams of hexamethyldisilizane (HMDS) had been previously added. The Cab-o-sil was added slowly with the Osterizer running at its lowest speed to minimize aerosolization of the Cab-o-sil. This powder, containing 40 wt. % of HMDS, was then ready for coating of powders.

The powders to be coated were divided into four samples. One was retained as a control sample and nothing was done to it. The second sample was placed in the Osterizer and stirred for two minutes. The third sample (10 grams of powder) was placed in the Osterizer with sufficient untreated Cab-o-sil that the 0.5 grams of Cab-o-sil formed a mixture containing 4.8 wt. % Cab-o-sil. This mixture was stirred for two minutes. Ten grams of the fourth portion of the powder were placed in the Osterizer with 0.86 gram of HMDS treated Cab-o-sil and this mixture was milled at low speed for two minutes. This mixture then contained 4.7% Cab-o-sil, which was essentially the same amount as was added to the third batch of powder.

During this powder preparation work it appeared that uncoated Cab-o-sil easily became charged and as a result tended to stick to the glassware in which it was stored. HMDS treated Cab-o-sil, however, did not appear to pick up the charge as easily.

Some of the basic properties of the powders used in these studies are recorded in Table I, including values of apparent density, crystal density, mass median diameter, and wt. % of powder 16 microns in diameter or less. The cornstarch and glycine powders have similar mass median diameters, and apparent densities, whereas the resorcinol has a lower density and a higher mass median diameter. The particle size distributions of these three powders are shown in Figure 1. Measurements of the dielectric constant and electrical conductivity of the cornstarch and glycine powder made under Contract No. DAAA-15-67-C-0509 (Ref. 5) indicate that glycine would be able to leak off charges which build up on it considerably more rapidly than cornstarch.

These powders, with different substrates, were tested for disseminability using a GCA elutriator and the MRC Reaerosolizer (Ref. 6). The GCA elutriator should be a valid test

Table I

PROPERTIES OF POWDERS

	CS	Corn Starch	Glycine	Resorcinol	Saccharin
Apparent Density, g/cc	0.25	0.64	0.76	0.44	0.65
Crystal Density, g/cc	1.4	1.51	1.58	1.27	1.58
Mass Median Diameter,	13.5	11.6	9.1	17.3	14.0
Wt % of Powder, 10 μ or less in diameter	33	38	56	32	32
Wt % of Powder, 30 μ or less in diameter	87	98	98	82	86

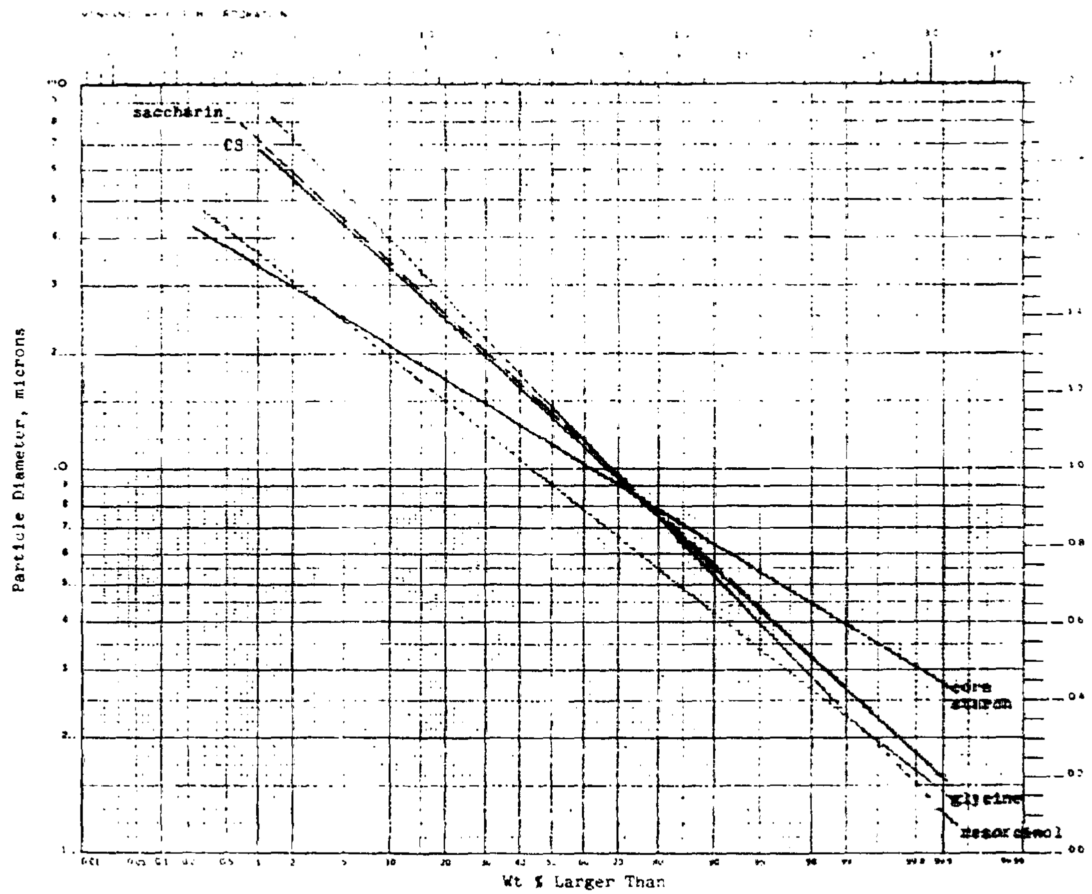

Figure 1. Particle size Distribution of CS, Cornstarch, Glycine, Saccharin and Resorcinol Powders Used in the Substrate Effects Study

for pneumatic disseminability since Edgewood data on GCA elutriation and the Mity Mite test on the same powders had a very high correlation coefficient (Ref. 6).

The results of the GCA elutriation tests on these powders are recorded in Table II for the "as received," milled, treated with Cab-o-sil, and treated with HMDS plus Cab-o-sil forms. The averages of the values quoted in Table II and their standard deviations (in parenthesis) are listed in Table III.

Examination of the data in Table III shows that there is a considerable range of percent of powder aerosolized in the "as received," with Cab-o-sil, and with HMDS-treated Cab-o-sil conditions. Some of these differences are highly significant statistically, as in the case of CS and resorcinol samples that have been treated with HMDS-treated Cab-o-sil. The difference between these averages is significant at greater than the 99% confidence level. The difference in means between cornstarch and glycine treated with raw Cab-o-sil is significant at the 95% confidence level. Many of the other differences in the table are also significant at the 95% level and additional ones could be proven at this level if more samples were run.

The significance of these results is that they show that the coatings of Cab-o-sil and HMDS-treated Cab-o-sil applied to these specific substrate particles in this specific manner did not completely mask the effect of the substrate particles. Although large differences in particle size distribution and particle density of the substrate particles could be expected to influence the terminal settling velocity of these treated particles, the differences of these properties in the samples the substrate particles used are not large enough to explain the differences observed. Calculations based upon a parabolic velocity profile inside the GCA elutriator indicates a velocity along the center line such that it would be expected to entrain all particles 30 microns or smaller in diameter. The relatively small difference in percentage of particles in the HMDS-treated Cab-o-sil samples of CS and resorcinol are insufficient to explain the sizable differences in percent aerosolized for the two samples. The fact that these two samples do perform so differently could conceivably be caused by an incomplete coating of the substrate particles. Further investigation to determine the cause is planned.

Table II

GCA ELUTRIATOR DATA ON SAMPLES WITH VARIOUS SUBSTRATE PARTICLES

Substrate Particles	Condition	Amount Aerosolized (wt %)
Cornstarch	As Received	31.4,21.8,16.9,19.2,18.7,20.1,16.7
	Milled 2 minutes	----
	With Cab-o-sil	6.6,13.2,13.4,16.6
	With HMDS treated Cab-o-sil	16.7,12.2,27.5,21.6,27.0
CS	As Received	22.7,16.8,18.0
	Milled 2 minutes	----
	With Cab-o-sil	20.3,23.4,32.3
	With HMDS treated Cab-o-sil	41.8,38.7,42.6
Glycine	As Received	21.3,20.9,16.8,16.8
	Milled 2 minutes	----
	With Cab-o-sil	25.0,36.7,19.7,39.7
	With HMDS treated Cab-o-sil	33.9,38.0,46.6,41.4
Resorcinol	As Received	7.9
	Milled 2 minutes	6.1
	With Cab-o-sil	9.8
	With HMDS treated Cab-o-sil	15.8,17.6,12.1,16.4
Saccharin	As Received	8.5,15.5, 8.1, 8.5,13.0
	Milled 2 minutes	----
	With Cab-o-sil	17.7,16.5,20.6
	With HMDS treated Cab-o-sil	24.4,25.2,33.5,33.4,33.2

Table III

SUMMARY OF RESULTS OF GCA ELUTRIATION TESTING OF SAMPLES WITH VARIOUS SUBSTRATE PARTICLES

Substrate Particles	Average Degree of Aerosolization (w/o) and Standard Deviation			
	As Rec'd	Milled 2 min.	With Cab-o-sil	With HMDS treated Cab-o-sil
Cornstarch	20.7(5.0)	-	12.4 (4.2)	21.0 (6.6)
CS	19.2(3.1)	-	25.3 (6.2)	41.0 (2.0)
Glycine	19.0(2.5)	-	30.2 (9.5)	40.0 (5.4)
Resorcinol	7.9	6.1	9.8	15.5 (2.4)
Saccharin	10.7(3.3)	-	18.2 (2.1)	29.9 (4.7)

Table III shows a general increase in percent aerosolization for basic particles treated with Cab-o-sil and a further increase in aerosolizability for particles treated with HMDS-treated Cab-o-sil. The only exception to this trend noted was for the case of treating cornstarch, where degree of aerosolization decreased from 20.7 to 12.4%.

MRC reaerosolizability tests were also run on these same powders. The data from these runs are recorded in Table IV. Averages of the data in Table IV and the standard deviations (in parenthesis) are shown in Table V.

The standard deviations on the reaerosolizability data shown in Table V are in general lower than the values for GCA elutriation in Table III. Therefore, it is easier to show statistical significance for differences between the averages listed. Because the standard deviations are so low, almost all of the averages listed in this table can be statistically proven to be different from the others. As a consequence, it is easy to show that these substrate powders all behave differently when treated with raw Cab-o-sil or the HMDS-treated Cabo-sil. It is interesting to note that upon comparison of the data in Tables III and V, that CS and glycine samples tended to give the highest results when treated with either Cab-o-sil or HMDS-treated Cab-o-sil and that the rescrcinol powders tended to give low results in all of these cases. This would indicate that the agglomeration forces acting during the performance of both of these tests affected the powders similarly, although of course, the percent of powder reaerosolized was, in most cases, less than that aerosolized in the GCA apparatus, as has been previously observed.

As in the case with the GCA elutriation tests, treatment of the raw powders with Cab-o-sil increased the reaerosolizability in all cases except that of cornstarch. Treatment with HMDS-treated Cab-o-sil was effective in increasing reaerosolizability with all five powders tested. As in the case of the GCA elutriation tests, it is suspected that the large differences seen in reaerosolizability of these powders may be due to incomplete coating. Investigation of this point is important since establishment that the characteristics of the substrate particles do not significantly affect performance if they are properly coated with raw Cab-o-sil or HMDS-treated Cab-o-sil could help us know under what conditions these types of coatings could be successfully used with different substrate particles without loss of performance.

Table IV

MRC REAEROSOLIZATION DATA ON SAMPLES WITH VARIOUS SUBSTRATE PARTICLES

Substrate Particles	Condition	Amount Reaerosolized (%)
Cornstarch	As Received	12.3,5.7,7.4,8.4
	Milled 2 Minutes	----
	With Cab-o-sil	5.9,10.3,7.6
	With HMDS treated Cab-o-sil	14.3,12.5,18.0,17.9
CS	As Received	7.0, 6.8
	Milled 2 Minutes	----
	With Cab-o-sil	21.2,22.0
	With HMDS treated Cab-o-sil	38.2,40.0
Glycine	As Received	4.4, 4.0
	Milled 2 Minutes	----
	With Cab-o-sil	24.5,25.8
	With HMDS treated Cab-o-sil	51.2,46.8
Resorcinol	As Received	1.0, 1.5
	Milled 2 Minutes	----
	With Cab-o-sil	12.3,14.3
	With HMDS treated Cab-o-sil	14.3,13.4
Saccharin	As Received	4.1, 4.1
	Milled 2 Minutes	----
	With Cab-o-sil	23.2,14.3,23.0
	With HMDS treated Cab-o-sil	37.3,39.9

Table V

SUMMARY OF MRC REAEROSOLIZABILITY RESULTS ON SAMPLES WITH VARIOUS SUBSTRATE PARTICLES

	Amount Reaerosolized (%) and Standard Deviation			
Substrate Particles	As Received	Milled	With Cab-o-sil	With HMDS Treated Cab-o-sil
Cornstarch	8.4 (2.8)	-	7.9 (2.2)	15.6 (2.7)
CS	6.9 (0.1)	-	21.6 (0.6)	39.1 (1.3)
Glycine	4.2 (0.3)	-	25.1 (0.9)	49.0 (3.1)
Resorcinol	1.2 (0.4)	-	13.3 (1.4)	13.8 (0.6)
Saccharin	4.1 (0.0)	-	20.1 (5.1)	38.5 (1.8)

4. TASK II -- DEVELOPMENT OF QUALITY CONTROL TESTS

The objective of Task II is to develop quality control tests which will differentiate between good and bad samples of CS-2. Numerous quality control tests were developed for (1) reaerosolizability, (2) particle size, (3) particle density, (4) flowability, (5) hydrophobicity, (6) sintering, (7) agglomeration, and (8) chemical composition. Studies were also made of aerosolizability, electrical properties, and particle surface characteristics (Ref. 6-7). Additional studies have been made this quarter to (1) determine the effect of sample size on reaerosolizability, (2) measure the adsorbed water and silanol OH on CS alpha E-218-840T and CS-2 green 401, (3) determine the effect of moisture on reaerosolizability, (4) further examine the particle surface characteristics of samples, and (5) perform multiple correlation studies of the data previously collected.

4.1 Effect of Sample Size Upon Reaerosolizability

The MRC reaerosolizability test consists of depositing a sample of the powder to be tested on the surface of a glass frit (Corning 36060) which has a cross-sectional area of 25.6 square centimeters. Dry nitrogen is then passed upward through the frit at a rate of 200 cc/sec and the reaerosolized powder is collected on a filter paper at the top of the apparatus (Ref. 6). Preliminary results showed that the amount of reaerosolizability found by weighing the filter papers at the top of the apparatus varied depending upon the sample size used. It was decided to further investigate this effect.

Samples of various sizes of powders, CS-alpha E-218-84 P.T., CS-alpha 7-48-2, CS-2-17 P.T., CS-2-26 Northrop Carolina, and CS-2-28 Dropped Spheres were used in these tests. The data obtained in these tests are recorded in Tables VI through X and these data are plotted in Figures 2 through 6.

Examination of these figures shows that the percentage of the samples reaerosolized definitely tends to increase in all five samples as the sample sizes increase. The curves, however, do not have the same shape. The percentage reaerosolized seems to rise continuously and smoothly for samples CS-alpha 7-48-2 and CS-2-28 Dropped Spheres; the other three samples all show discontinuity in their curves. The sharp breaks seen in the curves for CS-alpha E-218-84 P.T., CS-2-18-17 P.T., and CS-2-26 Northrop Carolina occur just above 0.2-, 0.2-, and 0.3-gram sample weights, respectively. These

Table VI

REAEROSOLIZABILITY DATA FOR CS-ALPHA E-218-84 P.T. AS A FUNCTION OF SAMPLE SIZE

Sample Weight (g)	Reaerosolizability (%)
0.10	14.0
0.14	32.5
0.18	28.2
0.20	33.6
0.23	33.6
0.28	35.0
0.34	48.0
0.37	34.5
0.41	36.9, 38.0
0.54	35.9

Table VII

REAEROSOLIZABILITY DATA FOR CS-2-26 NORTHROP CAROLINA AS A FUNCTION OF SAMPLE SIZE

Weight (g)	Reaerosolizability (%)	
0.11	0.7	
0.30	0.2	0.9
0.31	1.4	
0.42	10.2	6.3
0.50	7.7	

Table VIII

REAEROSOLIZABILITY DATA FOR CS-2-17 P.T. AS A FUNCTION OF SAMPLE SIZE

Weight (g)	Reaerosolizability (%)					
0.04	7.3					
0.10	18.5					
0.11	10.6	12.0	14.2	14.2	15.5	15.9
0.13	13.7	14.0	26.0			
0.20	28.0	31.0				
0.21	47.5					
0.22	31.8	45.4				
0.24	50.8					
0.25	47.3	48.7				
0.27	52.5					
0.29	23.2					
0.30	53.4	54.2				
0.31	50.8					
0.32	52.5					
0.35	54.6					
0.36	55.5					
0.38	54.2					
0.40	43.3					
0.42	58.6					

Table IX

REAEROSOLIZABILITY DATA FOR CS-ALPHA 7-48-2
AS A FUNCTION SAMPLE SIZE

Weight (g)	Reaerosolizability (%)	
0.05	4.3	
0.07	6.5	8.4
0.10	4.1	
0.11	9.1	
0.12	5.7	9.0
0.13	20.4	
0.14	10.2	
0.15	6.0	6.6
0.17	9.0	
0.25	16.1	
0.26	13.4	
0.31	12.2	
0.51	22.0	

Table X

REAEROSOLIZABILITY DATA FOR CS-2-28 DROPPED SPHERES
AS A FUNCTION OF SAMPLE SIZE

Weight (g)	Reaerosolizability (%)
0.10	12.7
0.20	33.6
0.27	33.3
0.28	38.8
0.30	41.3
0.42	56.0

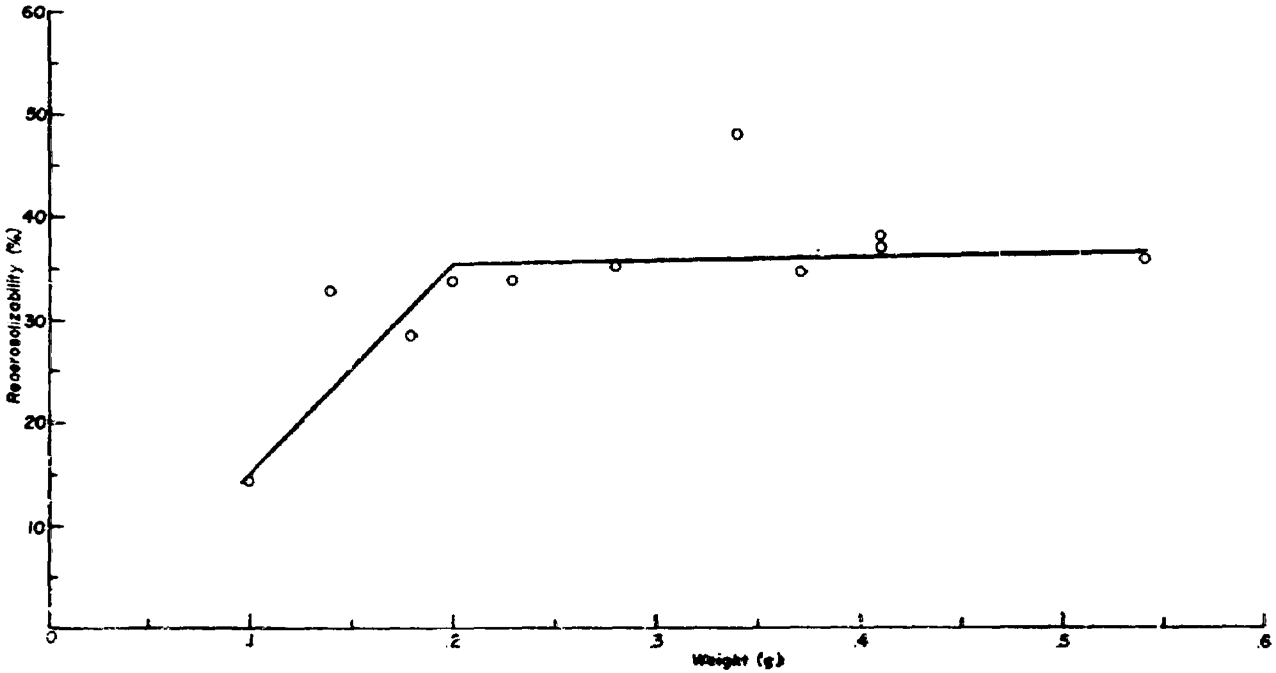

Figure 2. Reaerosolizability Data for CS-Alpha E-218-84 F.T. As A Function of Sample Size

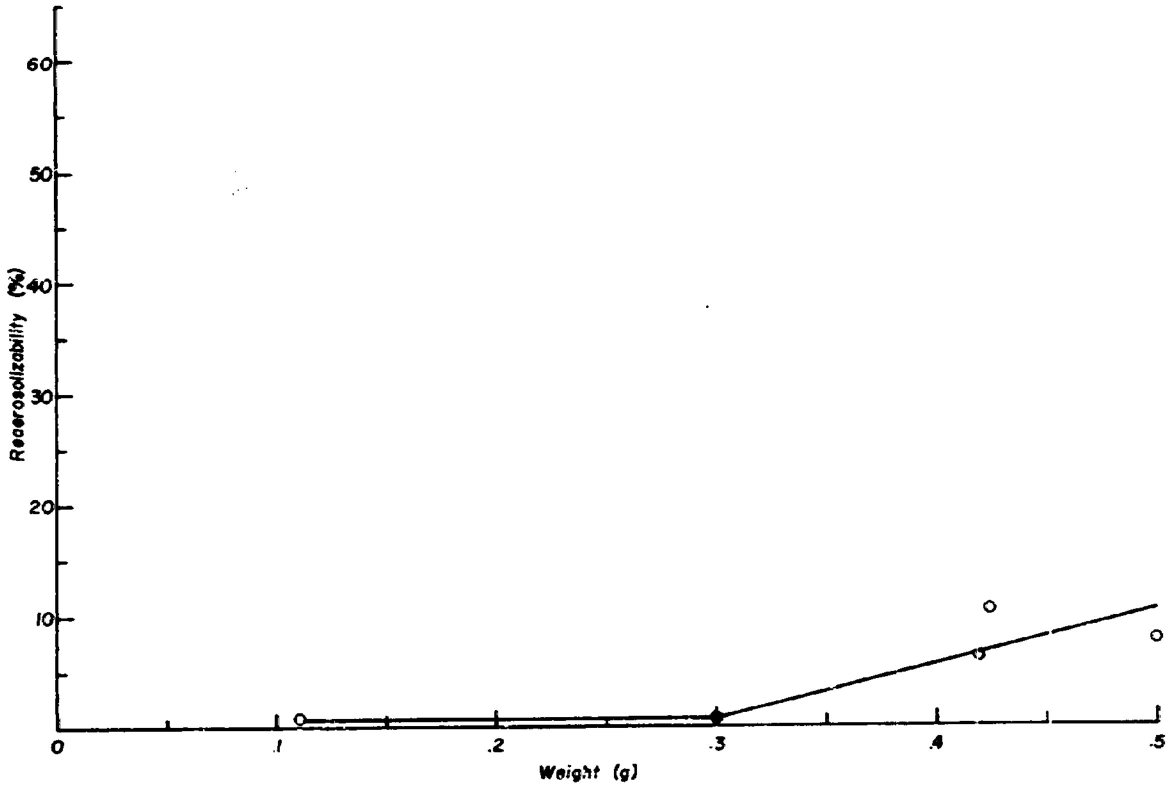

Figure 3. Reaerosolizability Data for CS-2-26 Northrop Carolina As A Function of Sample Size

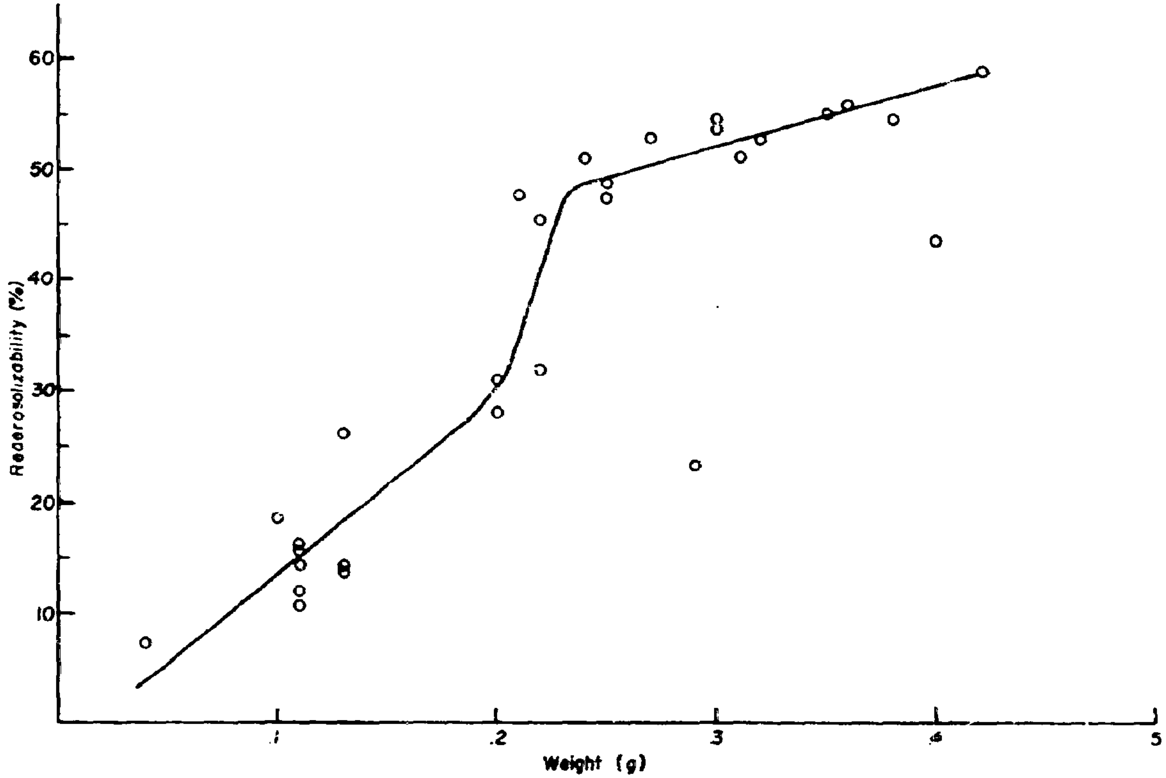

Figure 4. Reaerosolizability Data for CS-2-17 P.T. As A Function of Sample Size

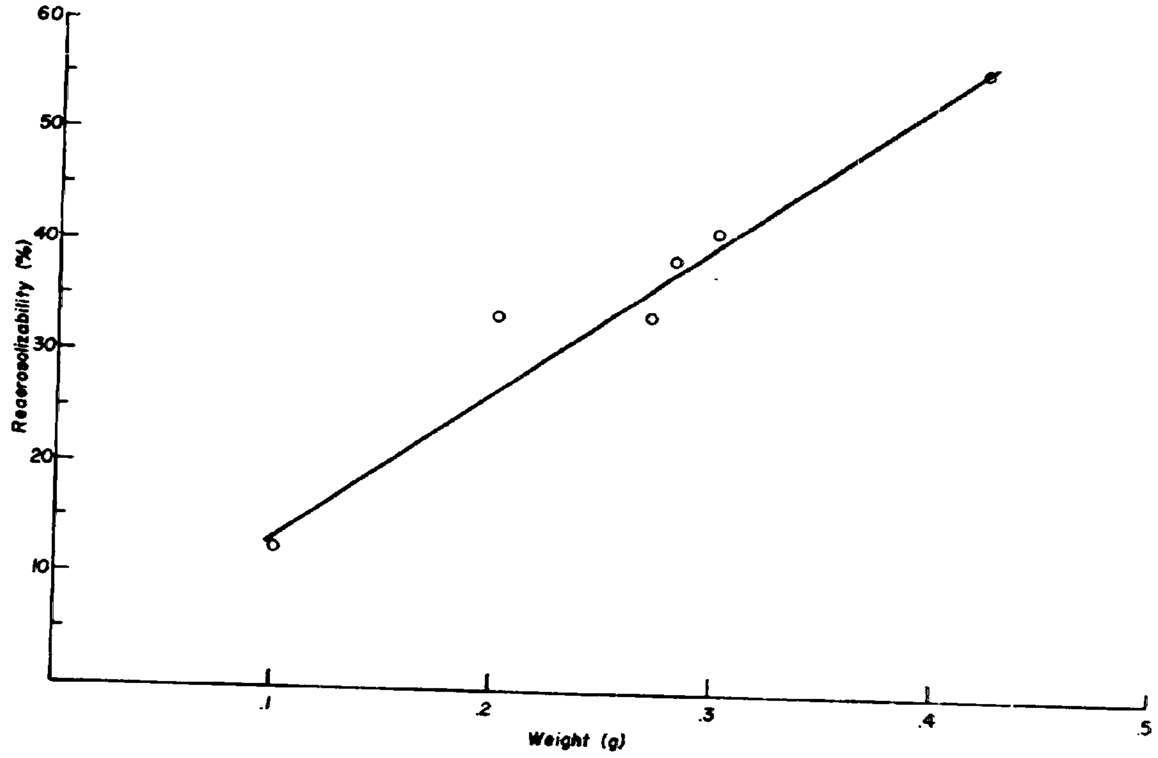

Figure 5. Reaerosolizability Data For CS-2-28 Dropped Spheres As A Function of Sample Size

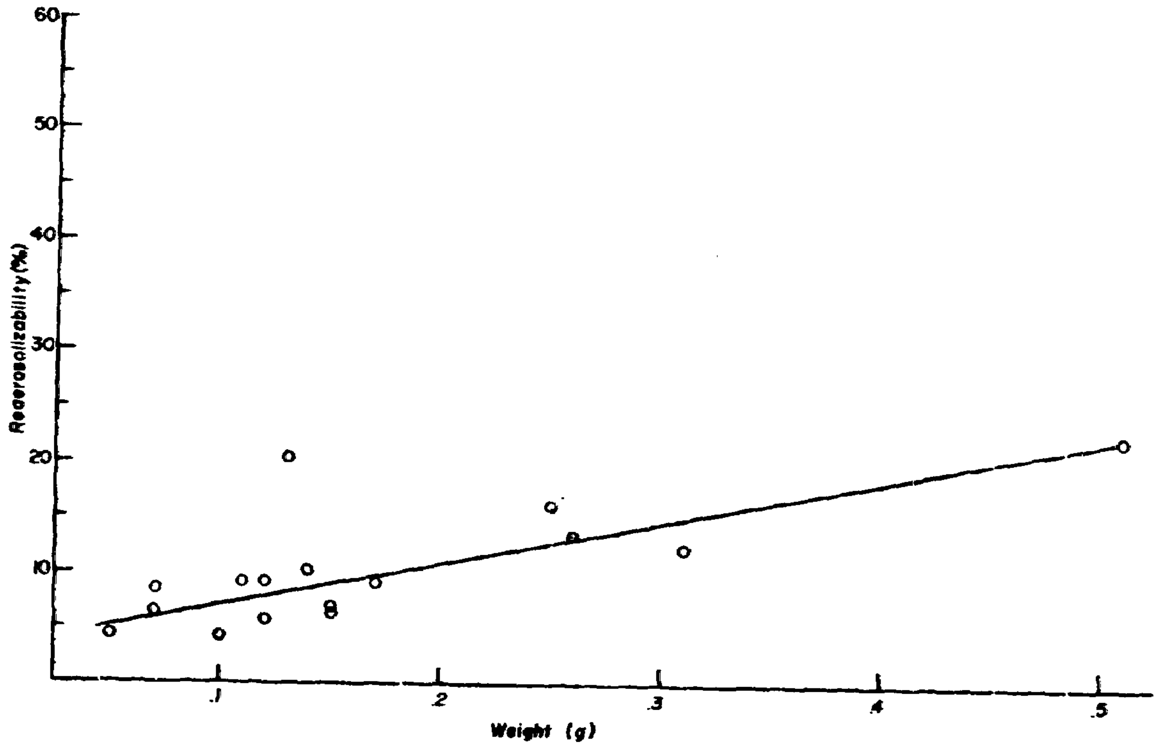

Figure 6. Reaerosolizability Data for CS-Alpha 7-48-2 As A Function of Sample Size

samples have apparent densities of 0.21, 0.22, and 0.30 gram per cc (Ref. 6). This then turns out to be a sample thickness of 0.040 centimeter for each of these samples, which is equal to a deposit of roughly 100 grams per square meter. That the reaerosolizability is connected to deposit thickness is reasonable based upon our observations of the reaerosolizability test. For thin samples the gas seems to channel between agglomerates of particles; whereas for thicker samples of the same material, the gas lifts the whole layer of powder and seems to reaerosolize a larger percentage of it when this layer breaks up and tries to settle.

It would appear from these data that a powder thickness exists at which sharply increased reaerosolizability may occur, but that this thickness, being close to 100 g/meter^2 (assuming an apparent density of 0.25 gram/sq. cm.), is so high as to be logistically undesirable for field use. At the lower levels of practical usage, the percent reaerosolizability seems to increase linearly with sample deposit thickness for all of the samples tested, although the slope is very low for CS-2-26 Northrop Carolina.

4.2 Chemical Analysis

The adsorbed water and silanol OH were determined on the additional two samples: CS alpha E-218-84 P.T. and CS-2 green #401 using the methods already outlined (Ref. 6).

Sample	% Total OH	% Adsorbed H_2O	% OH (H_2O)	% OH Silanol
CS alpha E-218-84 P.T.	0.19	0.070	0.13	0.06
CS-2 green #401	0.17	0.070	0.13	0.04

The results for the two materials are very similar and hence fail to supply any additional information as to the reason for the great difference in the reaerosolizability as measured by the MRC apparatus for the two samples. In general, the % OH silanol tends to be lower than most other samples of CS that have been studied whereas the amount of adsorbed water tends to be higher than most other samples. The total OH found for these two samples is very close to the average of 0.20% for all the samples studied.

4.3 Effect of Humidity on Reaerosolizability

Reaerosolizability measurements made last winter were noted to be a function of sample size used, but otherwise were constant for a given gas flow rate. When additional experiments were started this spring to further elucidate the effect of sample size upon reaerosolizability, erratic results were noted. Samples of CS-2-17 P.T. weighing o.28 and 0.34 gram had reaerosolizabilities of 49.0 and 43.4 percent, respectively, which agreed well with results obtained previously (Ref. 6).

Runs taken a few days later, however, with 0.16-gram samples yielded very low reaerosolizabilities, so a new sample was taken from the bottle. The bottle was rolled for one hour to produce uniform blending of the sample. Runs made on that day and the succeeding two days were consistent with the high values of reaerosolizability expected for this powder. On the fourth day, however, a value somewhat lower than expected was obtained and the high humidity present that day was suspected as being a possible cause. The results of these tests on CS-2-17 P.T. are summarized in Table XI.

The effects of humidity on reaerosolizability were also noted on samples of CS-2-28 dropped spheres. From our work on the study of reaerosolizability versus sample size, a sample of 0.30 gram of this powder would be expected to reaerosolize to approximately 40%. One sample of this material that had not been disturbed during the previous month was run using a 0.30-gram quantity and yielded a reaerosolizability of 29.4%. A sample of this powder that had been removed from the bottle the previous day and stored overnight in an open container exposed to the humid atmosphere yielded a reaerosolizability of 15.2% when tested at the same sample size. The next day the sample was rolled for one-half hour and a 0.30-gram sample gave results typical of those expected based upon our experience last winter. The following day, however, when this material was retested the reaerosolizabilities were measured as being only half as high. These data are recorded in Table XII.

Table XI

MRC REAEROSOLIZABILITY TESTS ON CS2-17 P.T.

Date	Sample Treatment	Sample Size, (g)	Reaerosolizability (%)
3-20-68	Not rolled during previous month	0.284	49.0
3-21-68	Not rolled during previous month	0.336	43.4
3-25-68	Not rolled during previous month	0.164	9.5
3-26-68	Not rolled during previous month	0.154	6.7
4-1-68	Rolled 4-1-68	0.353	54.6
4-2-68	Rolled 4-1-68	0.416	58.6
4-2-68	Rolled 4-1-68	0.362	55.5
4-3-68	Rolled 4-1-68	0.213	47.5
4-3-68	Rolled 4-1-68	0.303	54.2
4-4-68	Rolled 4-1-68	0.112	12.0
4-5-68	Rolled 4-5-68	0.115	15.9
4-11-68	Rolled 4-11-68	0.106	10.6

No variations of reaerosolizability with time or sample pretreatment were noted previously when runs were made in December and February. The present tendency for sample degradation with time seems to have started early in April, which coincides with the onset of higher humidity in Dayton.

Table XII

EFFECT OF SAMPLE TREATMENT ON REAEROSOLIZABILITY OF CS 2-28 DROPPED SPHERES

Date of Run	Sample Treatment	Sample Size (g)	Reaerosolizability (%)
4-29-68	Not rolled during previous month, sample exposed to humid air overnight	0.30	15.2
4-29-68	Not rolled during previous month, sample not exposed to humid air	0.30	29.4
4-30-68	Rolled 4-30-68	0.30	41.3
5-1-68	Rolled 4-30-68	0.31	21.6
5-1-68	Rolled 4-30-68	0.31	19.2

Our experience last winter indicated that powder in the bottles gave the same percent reaerosolizability when tested even weeks apart under conditions in which the bottle was opened periodically but in which the powder was not disturbed. This shows that the changes of reaerosolizability with time in April cannot be attributed to aging, especially since the temperature where the powders were stored did not change from winter through April. We, therefore, attribute the changes in the undisturbed powder that have occurred as being due to humidity since we know of no other factor relative to these samples that has changed between the April time period and the preceding winter. It is apparent from the data that the humidity caused increased agglomeration of these powders and that the percent reaerosolizability was dramatically affected. These effects were also observed visually during the testing. Samples that were exposed to moisture were seen to agglomerate on the glass frit to a much greater degree than those that were rolled to break up the agglomerates caused by humidity.

The values of reaerosolizability previously recorded for all of our powders were for samples in the dry condition and hence do not include the effect of agglomeration due to humidity. The fact that humidity does dramatically cause agglomeration is of considerable potential significance in this program, however, because humidity will limit the reaerosolizability and hence the time of denial factor (T_d) in the performance index. It appears that more information should be gathered concerning the quantitative effect of humidity upon reaerosolizability.

4.4 Particle Surface Characteristics - Electron Photomicrographs

A series of electron photomicrographs were presented in the Second Quarterly Report (Ref. 6) showing the appearance of a number of CS samples, both at a representative magnification and of a particular area of one sample at a number of magnifications. It was observed that the photographs showed various amounts of shiny and fuzzy particle areas in different proportions for different samples. It was suggested that this may be a result of the degree of coating of Cab-o-sil on the particles, although no apparent relationship was found that was related to the reaerosolizability of the particular sample.

These samples were prepared by coating an aluminum sample holder with rubber cement and then dusting some of the CS sample onto the cement. The sample was then given a coating of aluminum at most several angstroms thick by vapor deposition, which acts as a conducting surface to prevent charge buildup during the observation of the sample on the electron microscope. The shiny areas are a result of charge buildup at a site, which could occur when the surface was not completely covered with the conductive aluminum, or as a result of small particles being lost to expose some fresh sample that had not been ocvered by the aluminum. It appears to be unlikely that the aluminum coating would adhere more strongly to a treated Cab-o-sil or a CS particle covered with Cab-o-sil.

The difference observed and reported in the Second Quarterly Report most likely reflect the irregularity of the surface of the observed particles; a high irregular surface would be less completely coated than a smooth surface. As a result, the reported results would have little relation to the other measured properties of the CS materials.

A series of electron photomicrographs (Figures 7-14) have been obtained for samples before and after collection on the filter of the MRC reaerosolizability apparatus to determine if any significant change could be noted in the physical appearance as a result of reaerosolization. No apparent difference was observed for all the samples studied with the exception of a sample of CS-Alpha 7-48-2. The sample studied before reaerosolization showed a number of smooth spherical agglomerates with none of the coarse appearance seen for the remainder of the samples. After reaerosolization, the particles had an appearance similar to the other samples, but still as large agglomerates.

Another series of photomicrographs of CS-Alpha 7-48-2 (Figures 15, 16) were studied to check these results. In this case, the photographs before and after reaerosolization were very similar and much like the other CS samples studied. It would appear, therefore, that the original sample of CS-Alpha 7-48-2 was not typical of the powder and that the particular sample studied in the first series contained a high percentage of large smooth agglomerates. A more homogeneous sample of this material had the typical appearance of the other CS samples.

The electron photomicrographs of the repeat series on CS-Alpha 7-48-2 before and after reaerosolization at various magnifications are shown in Figures 7 and 8.

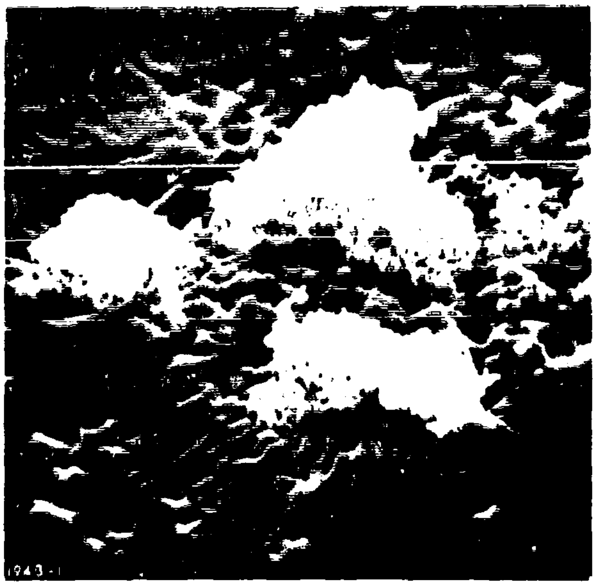

Figure 7 CS Alpha, Sample 1 -
As Received - 935X

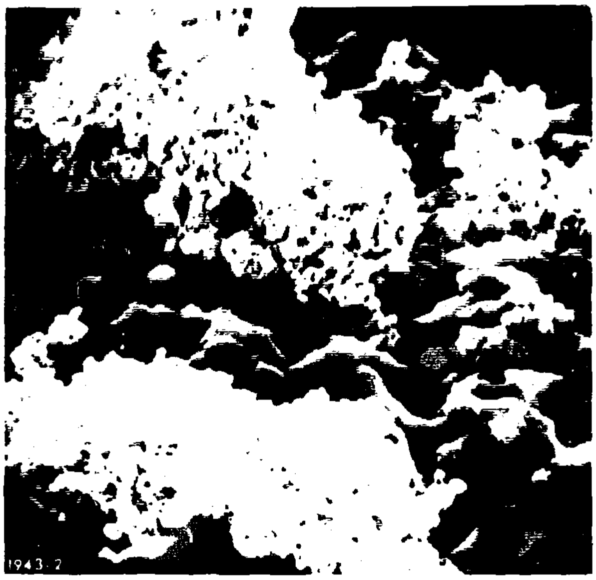

Figure 8 CS Alpha, Sample 1 -
As Received - at 1970X

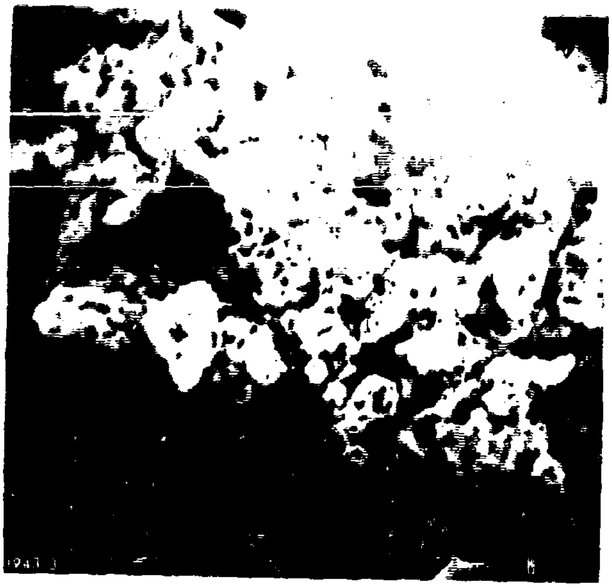

Figure 9 CS Alpha, Sample 1 -
As Received - at 4675X

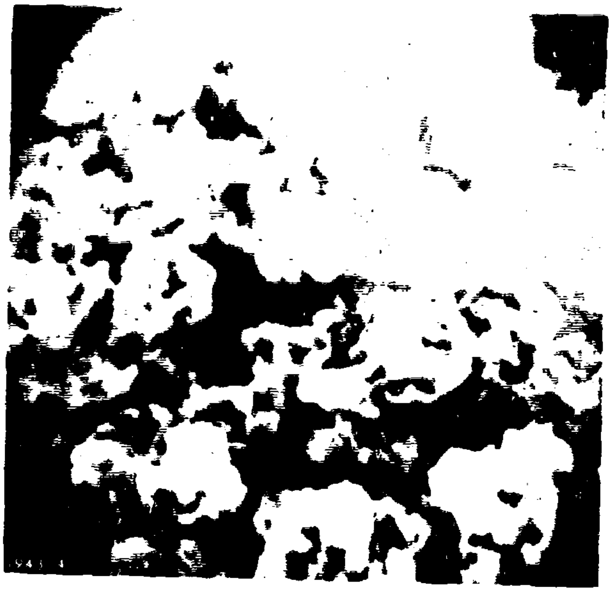

Figure 10 CS Alpha, Sample 1 -
As Received - at 9350X

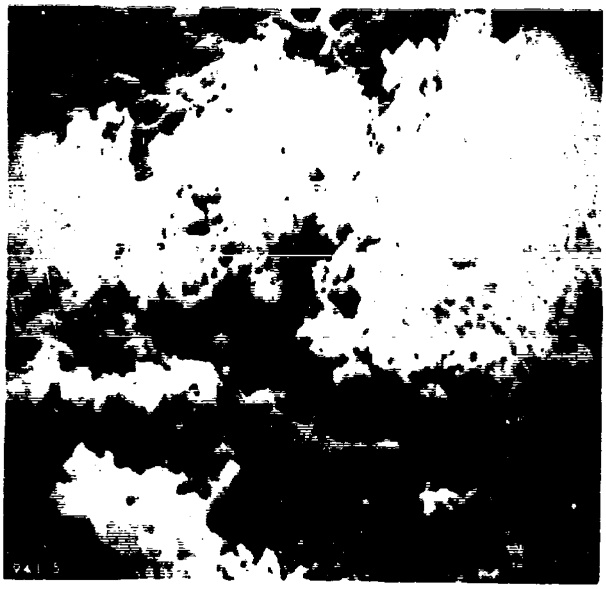

Figure 11 CS Alpha, Sample 1 - After Elutriation - at 935X

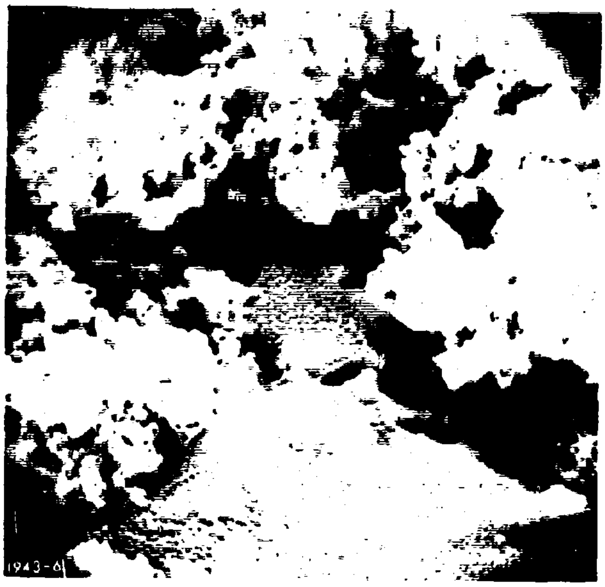

Figure 12 CS Alpha, Sample 1 - After Elutriation - at 1870X

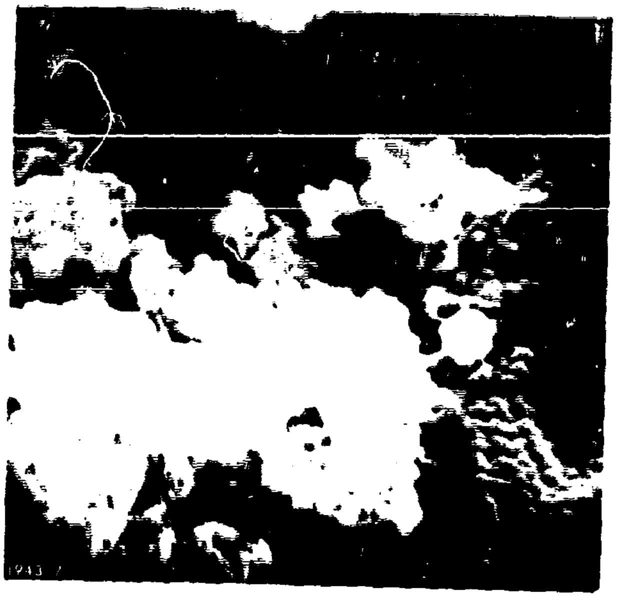

Figure 13 CS Alpha, Sample 1 -
After Elutriation -
at 4675X

Figure 14 CS Alpha, Sample 1 -
After Elutriation -
at 9350X

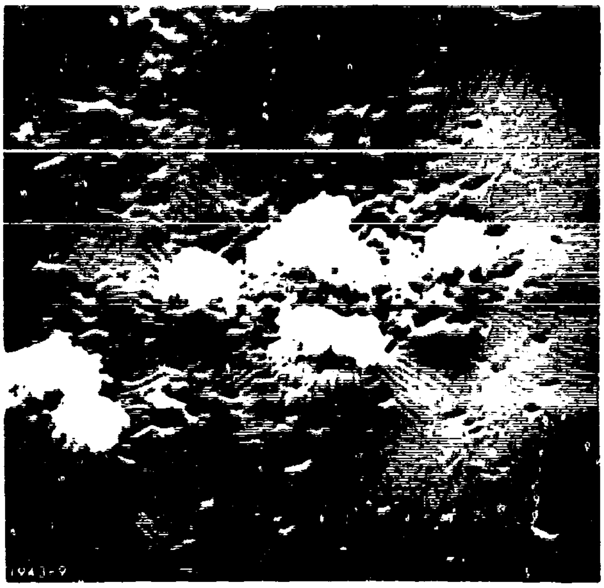

Figure 15 CS Alpha, Sample 2 - As Received - at 468X

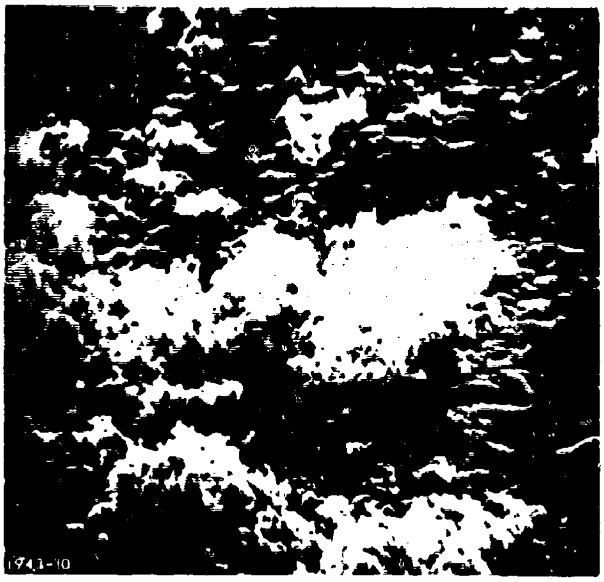

Figure 16 CS Alpha, Sample 2 - After Elutriation - at 468X

4.5 Multiple Correlation Analysis of Data

We have run multiple linear regressions using physical data on the thirteen CS formulations measured as a part of this contract (Ref. 6) to see if the results of the reaerosolizability test we have developed could be predicted as an algebraic function of these powder properties. The physical properties which were used as independent variables in these regressions were bulk density, fluid density, percent water, and hydrophobicity (Sparkleen). The regressions were effected by the backward elimination procedure (Ref. 8) in which coefficients are calculated for all of the terms of the proposed mathematical model followed by the sequential deletion of those terms whose calculated regression coefficients have the lowest significance as measured by the Student t-test. The program then selects as the best regression equation the one with the lowest residual variance.

A summary of the results of the various regressions we have tried is given in Table XIII. It can readily be seen from this table that using the logarithm of the reaerosolizability as the dependent variable in place of the reaerosolizability itself markedly improves the correlation coefficient of the best regression equation (e.g. the equation with the lowest residual variance) for the mathematical model being tested. This substitution also changes the residual variance by about three orders of magnitude. From the best equations resulting from each individual regression equations resulting from mathematical model 8 and 9 (which explain 78% of the variation in reaerosolizability) as the overall "best" regression equation. Although this is not the regression equation with the lowest residual variance, and highest correlation coefficients and percent variation explained, it was chosen on the basis of the following additional considerations. We insisted that since only 13 data points were used in the regressions that the "best" regression equation contain no more than four independent variables. Some of the other equations that were more mathematically favorable as far as overall closeness of fit were eliminated because they tended to oscillate widely for certain combinations of the independent variables.

Table XIII

REGRESSION ANALYSIS

	Variable	Mathematical Model*										
		1	2	3	4	5	6	7	8	9	10	11
1.	Bulk Density	us	r	u	r	u	us	rs	r	u	u	u
2.	Hydrophobicity	us	u	u	u	u	u	us	us	us	us	u
3.	Percent Water	u	u	rs	r	rs	us	u	rs	rs	us	r
4.	Fluid Density	u	r	us	rs	rs	u	rs	rs	rs	r	us
5.	[1]·[2]	p	ps	p	p	p	p	ps	p	p	ps	p
6.	[1]·[3]	ps	ps	p	ps	p	ps	ps	p	p	ps	p
7.	[4]·[2]	p	p	p	p	p	ps	ps	ps	ps	ps	ps
8.	[4]·[3]	ps	ps	p	p	p	ps	ps	p	p	ps	p
9.	Reaerosolizability	u	u	u	u	u	-	-	-	-	-	-
10	$\log_{10}$ [9]	-	-	-	-	-	u	u	u	u	u	u
	% Variation Explained	53.87	57.93	46.93	46.63	46.05	81.30	89.76	78.11	78.11	88.70	72.40
	Correlation Coefficient	.7340	.7611	.6851	.6827	.6786	.9017	.9474	.8838	.8838	.9418	.8508
	Residual Variance	226.0	183.3	208.0	209.2	211.5	.206	.1581	.2113	.2113	.1454	.2132

* u indicates that the first order variable listed was used; r indicates that the reciprocal of the first order variable was used; p indicates that the second order variable was used with first order variables or their reciprocals used to correspond with first order terms (e.g. variable 5 in mathematical model 2 contains the product of the hydrophobicity times the reciprocal of bulk density); s indicates that the variable appears in the regression equation with lowest residual variance.

The regression coefficients and values for the t-function for our overall "best" regression equation are given in Table XIV and the values predicted by it of reaerosolizability are compared to the observed values in Table XV. From studying these two tables, it is obvious that despite the overall accuracy of the predictions of this regression equation, there are some relevant independent variables missing. For example, the differences between the reaerosolizability of CS-2-Green #401 and CS-Alpha E-218-84 P.T. cannot be explained on the basis of the factors we have chosen to include in our mathematical models. We suspect that surface charge on the powder particles might be an important factor to be included.

Table XIV

REGRESSION COEFFICIENTS AND VALUES OF t-FUNCTION
OVERALL "BEST" REGRESSION EQUATION

(Mathematical Models 8 and 9)

Variable	Symbol	Regression Coefficient	t-Value
Hydrophobicity	H	0.7053	2.8156
[Percent Water]$^{-1}$	1/W	0.005323	1.0843
[Fluid Density]$^{-1}$	1/F	0.6211	3.1498
Hydrophobicity/ Fluid Density	H/F	-0.07714	2.2170
Constant	C	-4.613	--

$$\log_{10} R = -4.613 + 0.7053(H) + \frac{0.005323}{W} + \frac{0.6211}{F} - 0.07714\left(\frac{H}{F}\right) \qquad (2)$$

R = reaerosolizability

Table XV

OBSERVED VALUES OF REAEROSOLIZATION COMPARED WITH VALUES PREDICTED BY OVERALL "BEST" REGRESSION EQUATIONS (Mathematical Models 8 and 9)

CS Formulation	Observed (%)	Calculated (%)
CS-2-17 PT	48.0	44.06
CS-2-28Fisher	39.4	15.82
CS-2-28Undropped Spheres	38.9	39.40
CS-2-28 Dropped Spheres	36.1	39.97
CS-2-28-4	35.7	27.83
CS-BETA	27.7	5.57
CS-ALPHA 7-48-2	12.8	35.92
CS-2-26	11.0	12.52
CS-2-28 PT	9.0	12.32
CS-1-5	0.3	1.07
CS-2-26 Northrop Carolina	0.2	0.16
CS-2-Green-401	4.9	17.50
CS-ALPHA E-218-84 PT	48.0	17.50

5. CONCLUSIONS

1. The performance index suggested in this report offers a realistic measure of quality of performance for powders in terrain denial applications, and it should be measured on samples of powders in the field in order to provide a realistic quantitative basis for rating powders for this application.

2. The nature of the substrate particles used when coated with Cab-o-sil or with HMDS-treated Cab-o-sil by the method described in this report made a significant difference in the rating of the resultant powder by both GCA elutriation and MRC reaerosolizability tests.

3. The reaerosolizability of the powders tested in each case increased with increased sample size. In some cases (but not all) a sharp rise occurred in this curve, but this rise occurred at sample thicknesses that are too great to be of practical field significance.

4. Exposure to moisture reduces the reaerosolizability of CS-2 powders and hence would be expected to significantly affect their performance index.

5. Multiple correlation analysis shows there is a good correlation between the logarithm of reaerosolizability and reciprocal of percent water, hydrophobicity (Sparkleen), reciprocal of fluid density, and the rates of hydrophobicity to fluid density for the CS powders whose properties were precisely measured under this contract.

6. FUTURE WORK

Future work on this contract is expected to include:

1. Testing of the new types of coatings for Cab-o-sil to determine their affect on disseminability via GCA elutriation and MRC reaerosolizability tests.

2. Testing of the reaerosolizability of powders using dry air an place of dry nitrogen.

3. GCA elutriation and MRC reaerosolizability tests on samples that have been exposed to controlled humidity.

4. Determination of the reason that the substrate particles coated with Cab-o-sil and HMDS-treated Cab-o-sil differ considerably in disseminability.

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13. ABSTRACT The objectives of this program are 1 to develop quality control tests for CS-2 and 2 to study the surface chemistry of powders to determine the factors that limit their dissemination by pneumatic and explosive means.

A performance index is suggested that should be a measure of how well a powder performs in terrain denial applications, and an experimental program has been designed to determine quantitatively the relative value of several powders for this application. The suggested approach should make it possible to determine the optimum values of reaerosolizability, hydrophobicity, and particle size for terrain denial powders.

Presized samples of CS, cornstarch, glycine, resorcinol, and saccharin were coated with Cab-o-sil and HMDS-coated Cab-o-sil from a single batch of the silica powder by blending in an Osterizer blender. The disseminability of these powders differed significantly, depending upon which substrate particles were used and whether the Cab-o-sil was HMDS-coated or not.

Tests on CS-2 powders showed that reaerosolizability in all cases increased with increasing sample size. Powders that had been exposed to humidity were considerably less reaerosolizable than those that were stored dry. Multiple correlation analysis of previous data shows a good correlation between the logarithm of reaerosolizability and the reciprocal of percent water, hydrophobicity, the reciprocal of fluid density, and the ratio of hydrophobicity to fluid density.

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14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Density						
Hydrophobicity						
Particle Size						
Dissemination						
Elutriation						
Aerosolizability						
Reaerosolizability						
CS characterization data						
Coating formulation						
Powder Technology						