

ENGINEERING EXPERIMENT STATION
of the Georgia Institute of Technology
Atlanta, Georgia



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FINAL REPORT

PROJECT NO. A-233

STUDIES AND INVESTIGATIONS OF
AGGLOMERATION AND DEAGGLOMERATION
OF SOLID PARTICLES

By

CLYDE ORR, JR., J. M. DALLAVALLE,
THOMAS E. STONECYPHER and W. J. CORBETT, III

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CONTRACT NO. DA-18-064-404-CML-88

CHEMICAL CORPS PROJECT: 4-04-14-021

CHEMICAL CORPS--FORT DETRICK
FREDERICK, MARYLAND

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JUNE 15, 1957

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I. SUMMARY

The cohesiveness of powders, particularly dry bacterial materials was studied. Two types of tests, one the actual deagglomeration of powder masses and the other the tensile strength of powders, were conducted. This report presents equipment information, test procedures, analytical methods and results for these tests.

Deagglomeration was studied by blowing powders with a series of gas pressures through capillary tubes of various diameters and lengths. Particle size distributions were determined after passage through the tubes and the velocity of powder movement was determined by high-speed photography. From the median particle size which resulted and the characteristics of the flow, conclusions about the process were drawn. As expected, greater tube length and smaller tube diameter gave increased dispersion. For a particular tube, increasing the Reynolds number (the ratio of inertial and viscous forces) increased the degree of dispersion.

Deagglomeration was also carried out by blasting powders through an annular port at various pressures in order to obtain rapidly an indication of the dispersion qualities of a number of powders. These tests, in general, showed that washing increases the dispersibility of bacterial powders. Grinding seemed to have less influence on ease of dispersion, though, of course, it permitted smaller size particles to be generated.

By forming a bed of powder on a device one part of which moved on precision ball bearings, the powder bed could be pulled apart and the force required could be measured. The tensile strengths of powders were obtained in this manner under rigorously controlled humidity conditions. Data were

obtained for powders under various degrees of compaction. In agreement with the deagglomeration-through-tube tests, the results showed that washing generally reduced the tensile strengths of bacterial material. Little other correspondence between the two types of measurement is evident. This result is probably due to the presence of both loose and tightly-compacted aggregates in any mass of powder. In dispersion tests the tight aggregates control the result while in bulk tensile strength tests the loose aggregates are the limiting factor.

The tests and apparatus used in the final stages of this investigation are recommended as the best now available for evaluating the cohesiveness of powders.

II. INTRODUCTION

The basic objectives of this project were to determine the nature and magnitude of forces holding solid particles together in agglomerates and to study mechanisms whereby these forces could be overcome and agglomerate-free aerosols produced.

The first year's progress was documented in a semifinal report¹ which presented a survey of the pertinent literature, theoretical studies, and experimental investigations bearing on the nature of forces between particles. This report covers the second and final year of the investigation. Specific techniques and instrumentation for cohesive-force and deagglomeration measurements and values applicable to dry bacteriological materials are its principal subjects, but the conclusions and recommendations presented later are drawn from the results and experiences of the two years.

For completeness the contract Statement of Work is reproduced here.

A. Scope

The contractor, as an independent Contractor and not as an agent of the Government, shall furnish the necessary services, personnel, labor, materials, tools, machinery and equipment, facilities and supplies not furnished by the Government, and do all things necessary for the conduct of studies, experimental investigations and tests to determine the nature and influencing factors of forces holding solid particles together in agglomerates, and mechanisms whereby these forces can be broken down and agglomerate-free aerosols

- - - - -
1. Orr, C. Jr., DallaValle, J. M., et al, Studies and Investigations of Agglomeration and Deagglomeration of Solid Particles. Semifinal Report, Project No. A-233, Engineering Experiment Station of the Georgia Institute of Technology, Atlanta, Georgia, June 30, 1956.

produced. In performance of the aforementioned work and services, the Contractor shall:

1. Conduct a survey of pertinent literature to:

(a) Determine the present state of knowledge on the mechanism and nature of agglomeration and deagglomeration of solid particles; and

(b) Review the various techniques used to disseminate dry particles as an aerosol, and determine to what extent the deagglomerating mechanisms involved in these techniques are fully understood, and whether or not such mechanisms are feasible for use and further study under this contract.

2. Conduct a study of the mechanisms of deagglomeration of solid particles ranging in size from 0.1 micron to 20 microns and of various compositions, including biological materials furnished by the Government. This study will include:

(a) An analysis of the laws governing particles in contact, under which the following forces will be treated, consideration being given to such aspects as the nature of the forces (such as electrostatic, van der Waal, chemical, and similar forces), their magnitude and direction, and factors affecting them (such as size, shape, composition, moisture content, temperature, and other pertinent factors):

(1) cohesive forces between particles

(2) frictional forces between particles and agglomerates.

(b) An analysis of the forces required to overcome cohesive, frictional and inertial forces, such analysis to include:

(1) size, shape, mass, and composition of the agglomerate;

and

(2) size, shape, mass, and composition of the individual particle.

(c) A theoretical analysis of methods capable of developing deagglomerating forces, and of the statistics and mechanism of recombination as a result of particle collision; such methods to include, without limitation:

- (1) compressible flow
- (2) incompressible flow-compressible flow combination, and
- (3) electrostatic, chemical, ultrasonic.

3. Test the various theoretical hypotheses at significant stages in the development of the analyses, and establish scaling laws for the various mechanisms under study; to include, without limitation:

(a) particle size and mass concentration measurements of aerosols produced by the various deagglomeration mechanisms;

(b) measurement of the size of agglomerates and numbers of basic particles comprising agglomerates which exist, both in aerosol form and as "fall-out" due to physical decay; and

(c) performance of deagglomeration - viability screening tests with biological simulants, and the monitoring of similar tests as may be conducted by Government personnel at Fort Detrick relative to the work and services called for under this contract.

4. Recommend to the Government:

(a) Specific techniques and instrumentation which may be applicable to routine cohesive force measurements of dry biological materials under controlled conditions of relative humidity.

(b) Techniques and instrumentation for the measurement of other physical properties of fine powders which the Contractor considers important in the problem of agglomeration and deagglomeration of solid particulates."

III. INVESTIGATIONS AND RESULTS

A. Capillary Deagglomeration

1. General

The dispersion of particulate materials upon being blown through capillary tubes by compressed gas was given a large share of the experimental study. A slightly modified Micromerograph, an instrument manufactured by the Sharples Research Corporation, Bridgeport, Pennsylvania, was employed. The deagglomerating mechanism of this device was replaced by a holder in which could be mounted a series of capillary tubes of different diameters and lengths. This change permitted a powder sample to be blown through a tube and analyzed in a relatively short time for size distribution.

A brief explanation of the Micromerograph may be in order. Basically it consists of three parts: (1) a deagglomerating section by means of which a dispersed sample is generated as a cloud of particles, (2) a settling column in which the particles are separated according to size by falling under the influence of gravity, and (3) an electronic balance and recorder to give the mass of powder accumulated at the bottom of the settling column as a function of time. The deagglomerator, as described above, was replaced by a capillary tube in this investigation. Auxiliary parts of this section were a sample holder, pressure indicators, a small compressed-gas tank and a quick-acting solenoid valve. The settling section was a 8-cm diameter, 220-cm long column vertically mounted. The deagglomerator mechanism was located at its upper end and the electronic-balance pan was mounted below its lower end. The recorded mass-time curves were reduced to particle size distributions with the aid of templates prepared in part from calibrating tests with materials of known distributions.

Capillary deagglomeration from the viewpoint of dimensional analysis and the fluid flow characteristics of the modified dispersing system are treated in following parts of this section.

2. Dispersion Analyses

a. Dimensional Analysis. In order to relate the variables believed to govern deagglomeration of particulate materials flowing in small tubes, a procedure known as dimensional analysis was used. This procedure requires that the controlling variables be known or assumed. Those considered to be important for the particular system of this study are given in Table I.

TABLE I
VARIABLES USED FOR DIMENSIONAL ANALYSIS

Variable	Symbol	Units
Tube Diameter	D	L
Tube Length	L_T	L
Fluid Mass Flow Rate	G	M/L^2T
Fluid Viscosity	μ	M/LT
Particle Density	ρ	M/L^3
Measured Mass Mean Diameter	D_p	L

For a relation between these variables to apply, a function

$$\phi(D, L_T, G, \mu, \rho, D_p) = 0 \quad (1)$$

must exist. Further, the fundamental units of mass, length and time (M, L, T) must be defined in terms of three incompatible variables. Thus,

$$D = L, \quad (2)$$

$$G = M/L^2T, \text{ and} \quad (3)$$

$$\rho = M/L^3. \quad (4)$$

The three variables given above are said to be incompatible when no combination of any two or three will give a dimensionless group. Equations 2, 3, and 4 expressed in terms of fundamental M, L, T units are

$$L = D, \quad (5)$$

$$T = \rho D/G, \text{ and} \quad (6)$$

$$M = \rho D^3. \quad (7)$$

Each of the remaining variables (L_T, μ, D_p) will also give a dimensionless "π" terms:

$$L_T = L = D; \pi_1 = L_T/D, \quad (8)$$

$$\mu = M/LT = \frac{\rho D^3 G}{D \rho D}; \pi_2 = \frac{\mu}{DG}, \text{ and} \quad (9)$$

$$D = L = D; \pi_3 = D_p/D. \quad (10)$$

Equation 1 can now be written

$$\phi(\pi_1, \pi_2, \pi_3) = 0, \quad (11)$$

or in dimensionless terms

$$\phi\left(\frac{L_T}{D}, \frac{\mu}{DG}, \frac{D_p}{D}\right) = 0. \quad (12)$$

In many cases, a correlation of the various dimensionless groups may be obtained by an equation of the form

$$\pi_1 = \alpha (\pi_2)^a (\pi_3)^b, \quad (13)$$

where α , a , and b are constants. If a correlation exists, the values of the constants can be obtained from experimental data.

b. Micromerograph Flow System Analysis. The two-phase, unsteady-state, compressible flow system of the Micromerograph as used in this investigation is shown schematically in Figure 1.

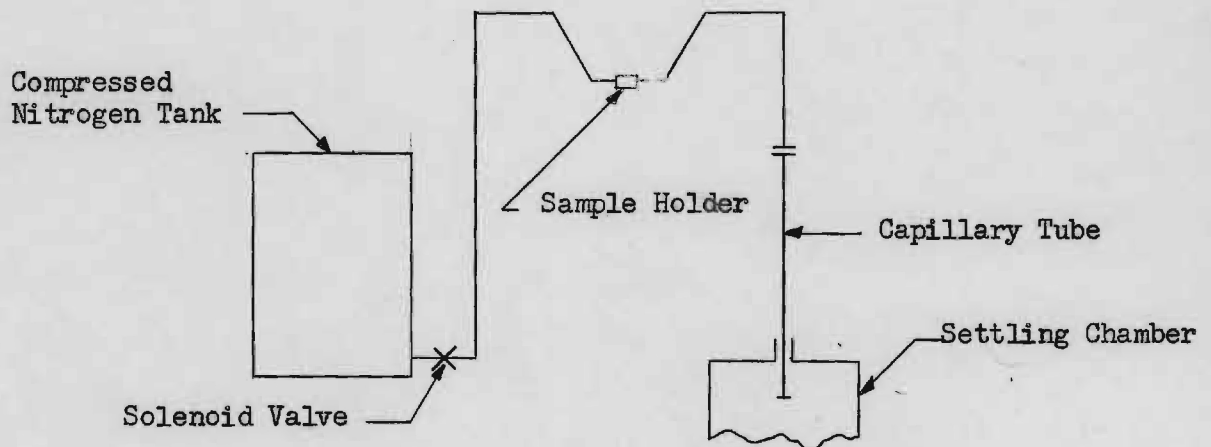


Figure 1. Micromerograph Flow System.

The method by which the system was treated is analogous to that given by Lapple². Writing an overall material balance on the tank gives

$$dn = - \left(\frac{G}{G_{cni}} \right) G_{cni} A \left(\frac{454}{M} \right) d\theta, \quad (14)$$

where n = number of gram-moles in tank,

G = mass flow rate, $\text{lb/ft}^2 \text{ sec}$,

2. Lapple, C. E., Fluid and Particle Mechanics, Edwards Brothers, Incorporated, Ann Arbor, Michigan, 1951.

G_{cni} = critical isothermal nozzle flow rate, lb/ft² sec,

A = area of pipe, ft²,

M = molecular weight of gas, and

θ = time in seconds.

By definition

$$G_{cni} = P \left(\frac{g_c M}{e R T} \right)^{1/2}, \quad (15)$$

where P = pressure, lb/ft²,

g_c = gravitational constant, ft/sec²,

e = base of natural logarithms,

R = gas constant, and

T = absolute temperature.

Substituting equation 15 into equation 14, and simplifying,

$$dn = -C \left(\frac{P}{T^{1/2}} \right) d\theta, \quad (16)$$

where

$$C \equiv \left[\frac{G}{G_{cni}} A \frac{454}{M} \right] \left(\frac{g_c M}{e R} \right)^{1/2}. \quad (17)$$

From the perfect gas law,

$$n = \frac{PV}{RT}, \quad (18)$$

and

$$dn = \frac{PV}{RT} \left[\frac{dP}{P} - \frac{dT}{T} \right], \quad (19)$$

where V = volume of tank.

Assuming adiabatic and reversible expansion in the tank,

$$T = T_o \left(\frac{P}{P_o} \right)^{\frac{k-1}{k}}, \quad (20)$$

where T_o = original absolute temperature in tank,

P_o = original pressure in tank, and

k = ratio of heat capacities of the gas.

Substituting equation 20 into equation 19 and simplifying yields

$$dn = \frac{n_o dP}{k (P_o)^{1/k} (P)^{\frac{k-1}{k}}} \quad (21)$$

where n_o = number of original gram-moles in tank.

Substituting equation 20 and equation 21 into equation 14 and simplifying gives

$$d\theta = - \frac{n_o T_o^{1/2}}{C k P^{\frac{k+1}{2k}}} \frac{dP}{P^{\frac{3k-1}{2k}}} . \quad (22)$$

Integrating equation 22 gives

$$\theta = \frac{2n_o T_o^{1/2} \left(P^{\frac{1-k}{2k}} - P_o^{\frac{1-k}{2k}} \right)}{C (k-1) P_o^{\frac{k+1}{2k}}} . \quad (23)$$

Thus, the time required for the tank pressure to fall from an original pressure P_o to a final pressure P can be readily calculated from a knowledge of T_o , k , n_o , and C .

The temperature T_0 was taken to be the temperature of the system; k for nitrogen, the dispersing gas, has a value of 1.4. The original number of moles in the tank, n_0 , can be calculated from the perfect gas law (equation 18) provided the volume of the tank is known.

Since the internal volume of the tank could not be measured directly without disassembling the apparatus, the equipment shown schematically in Figure 2 was used.

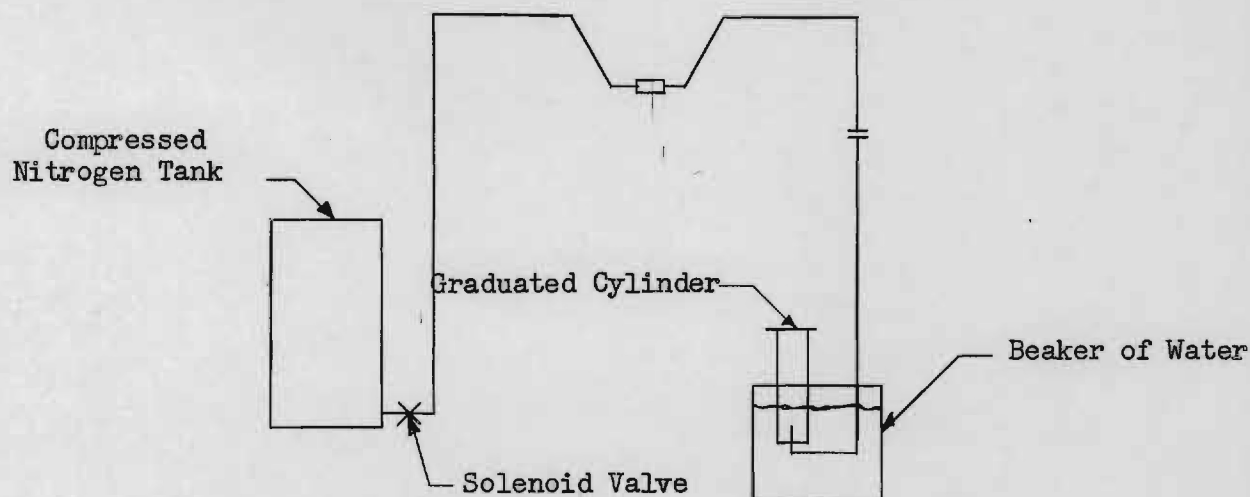


Figure 2. Equipment for Tank Volume Calibration.

The experimental technique involved charging the storage tank to a pressure, P_0 . A graduated cylinder filled with water was inverted in a beaker of water and the end of the tube was placed beneath the cylinder. The tank was then discharged into the cylinder, the volume of gas displacing water was determined at one atmosphere, and the volume of the tank was calculated by the relation

$$P_o V = (P - P_{H_2O}) (V + V_c), \quad (24)$$

where V = tank volume,

P = atmospheric pressure,

P_{H_2O} = partial pressure of water, and

V_c = volume of gas in graduated cylinder.

The data and calculated volumes are shown in Table II.

TABLE II
CALIBRATION OF GAS STORAGE CYLINDER

<u>Tank Pressure</u> (psig)	<u>Graduated Cylinder Volume*</u> (cm ³)	<u>Tank Volume</u> (cm ³)
100	187	26.1
100	190	26.5
50	95	26.4
50	92	25.5

* Measured at 21°C and 743 mm Hg. Vapor pressure of water = 18 mm.

The symbol C , as defined in equation 16, was evaluated from Figure 2, given by Lapple³. The parameter N used by that author, for the system under consideration, was also calculated by summing the number of equivalent velocity heads lost because of friction:

3. Lapple, op. cit., p. 61.

1 - globe valve	6.3
2 - 90° elbows	2.6
4 - 45° elbows	1.2
1 - abrupt entrance	0.5
Tube and powder	<u>1.4</u>
	12.0

Thus, for a particular pressure ratio $\frac{P}{P_0}$ the value of $\frac{G}{G_{cni}}$, the only variable affecting C , can be evaluated, and calculations of the length of time required to discharge the tank to some final pressure can be made.

3. Experimental Methods

The experimental method employed was essentially the same as that used in the ordinary operation of the Micromerograph. To begin a series of tests, a capillary tube was selected and attached to the system in the location formerly occupied by the deagglomerator. The material to be investigated was placed in the powder holder, the settling column was sealed, the storage tank was charged to a particular pressure with dry, compressed nitrogen gas, the recorder was started, and the powder was dispersed by the nitrogen. The recorder then produced a time-mass deposit chart which was analyzed with the templates supplied with the instrument. After a series of pressures was used with a particular tube, another tube was installed and the procedure was repeated. The entire procedure was repeated with a number of different materials.

Another series of tests in these investigations was conducted in the same manner described above, except that no tube was attached to the flow system. This permitted an estimate of the effect of the flow system above on the degree of dispersion of the powder.

4. Experimental Results

Investigations of the dispersion of various powders upon being blown through capillary tubing were conducted as described above. The series of investigations with the 3.3μ Serratia marcescens material begun in the preceding year and reported previously⁴ was completed. The remainder of the results for this material is given in Table VIII in the Appendix; for these investigations, tubes of 0.5-inch length by 0.1875-inch, 0.082-inch and 0.0446-inch inside diameter were used. Two non-biological materials--cadmium oxide and sulphur--were also investigated, using nine tubes of 0.5-, 1.0-, and 2.0-inch lengths by 0.1875-, 0.0820-, and 0.0446-inch diameters. The results of these investigations are given in Tables IX and X in the Appendix. Investigations in which unground Bacillus subtilis and 8.7μ Serratia marcescens were dispersed through the Micromerograph channel only were conducted; the 8.7μ Serratia marcescens was also dispersed through nine tubes of 1.0-, 2.0-, 3.0-inch lengths by 0.070-, 0.042- and 0.020-inch inside diameters. Results of these investigations are given in Tables XI, XII and XIII, respectively, in the Appendix.

Since there was uncertainty that the powder was completely dispersed before the entire amount of dispersing gas had discharged, high-speed motion pictures of the process were taken. These results indicated, even in the most extreme case, that the time for passage of the powder through the tube was less than 0.05 second. A typical portion of one of these films is shown in Figure 3.

4. Orr, et al., op. cit., p. 160-7.

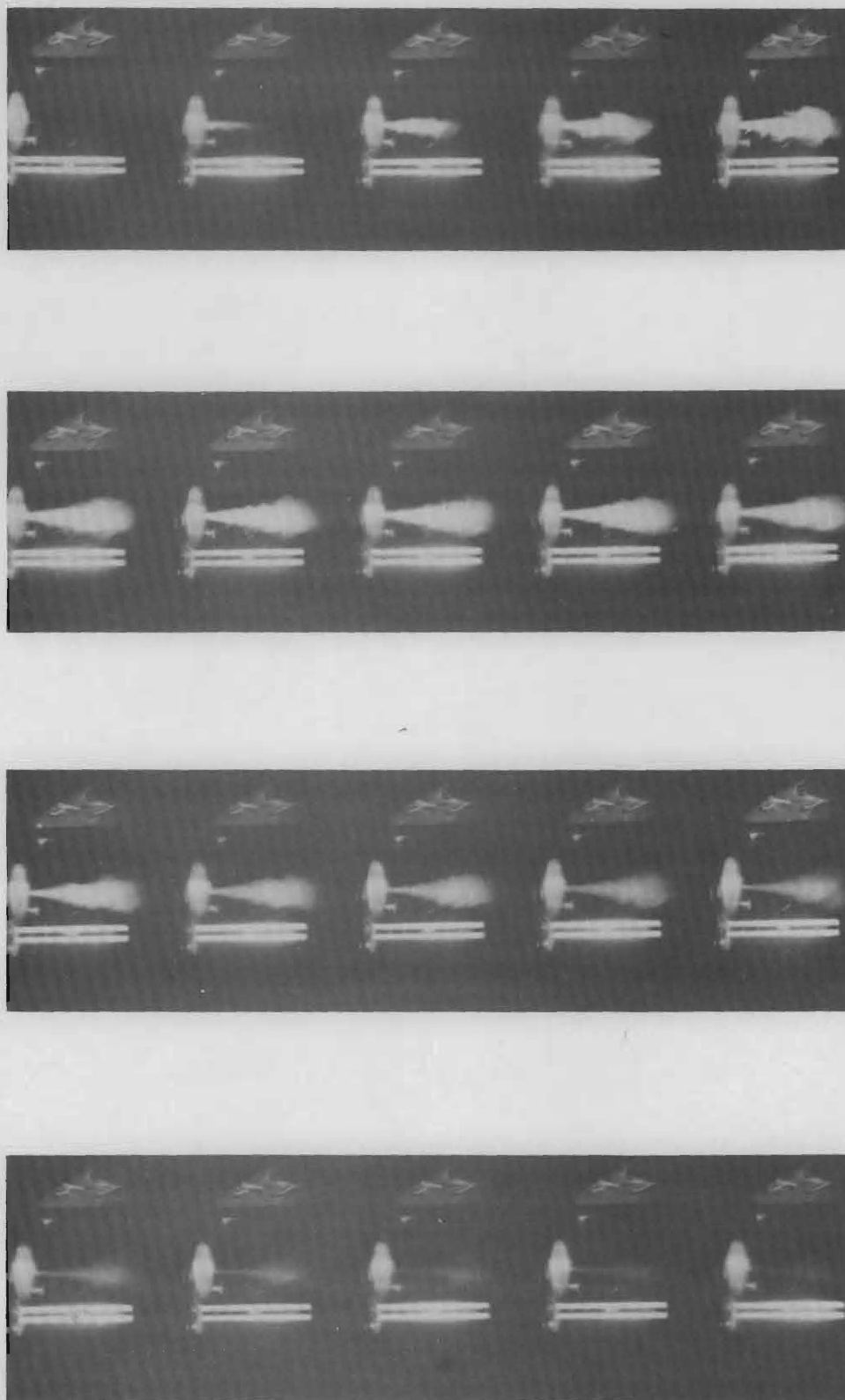


Figure 3. High Speed Photographs of Powder Discharging from a Capillary Tube.

From these studies, a typical time for powder dispersion was obtained. This time was used in equation 23 to calculate the pressure just after the powder passed. These calculations (see Appendix) indicated that the pressure at the end of dispersion was quite close to the initial pressure, and thus the powder was flowing through the tube at an essentially constant mass flow rate. Calculations were then made to obtain the Reynolds number--the ratio of inertial forces to viscous forces--as obtained in the dimensional analysis. Values for the Reynolds number as a function of tube diameter and initial pressure are given in Table III. Corresponding values of the calculated Reynolds number and the measured mass mean diameter of the dispersed powder are shown for various powders in Table IV.

TABLE III
REYNOLDS NUMBER FOR VARIOUS TUBE DIAMETERS AND PRESSURES

Pressure (psig)	Reynolds Number for Stated Tube Diameter					
	0.1875 in.	0.0820 in.	0.070 in.	0.0446 in.	0.040 in.	0.020 in.
400	1.57×10^6	6.85×10^5	5.85×10^5	3.74×10^5	3.51×10^5	1.67×10^5
300	1.19×10^6	5.22×10^5	4.45×10^5	2.84×10^5	2.67×10^5	1.27×10^5
200	8.15×10^5	3.56×10^5	3.04×10^5	1.94×10^5	1.82×10^5	8.66×10^4
100	4.35×10^5	1.90×10^5	1.62×10^5	1.03×10^5	9.70×10^4	4.63×10^4
60	2.83×10^5	1.24×10^5	1.06×10^5	6.74×10^4	6.34×10^4	3.02×10^4
50	2.45×10^5	1.07×10^5	9.15×10^4	5.84×10^4	5.49×10^4	2.61×10^4

B. Bulk Tensile Strength of Powders

Several devices for studying the tensile strength of bulk powders were constructed. Basic parts of all the devices were two steel blocks, machined so that their upper surfaces were level when the blocks were placed side by

TABLE IV

COMPARISON OF REYNOLDS NUMBER AND MEASURED
PARTICLE MASS MEAN DIAMETER

Reynolds Number and Particle Diameter for Stated Tube Diameter						
Tube Length (in.)	0.1875 in.		0.0820 in.		0.0446 in.	
	DG	D	DG	D	DG	D
	μ	p	μ	p	μ	p
		(μ)		(μ)		(μ)
<u>3.3μ Serratia marcescens</u>						
0.5	1.57×10^6	5.4	6.85×10^5	3.7	3.74×10^5	3.9
0.5	1.19×10^6	5.8	5.22×10^5	4.7	2.84×10^5	6.9
0.5	8.15×10^5	6.4	3.56×10^5	4.8	1.94×10^5	9.4
0.5	4.35×10^5	8.1	1.90×10^5	4.9	1.03×10^5	11.0
0.5	2.45×10^5	9.8	1.07×10^5	5.1	5.84×10^4	12.0
<u>Cadmium Oxide</u>						
0.5	1.57×10^6	2.9	6.85×10^5	3.0	3.74×10^5	3.1
0.5	1.19×10^6	3.3	5.22×10^5	3.4	2.84×10^5	3.2
0.5	8.15×10^5	2.8	3.56×10^5	3.5	1.94×10^5	4.3
0.5	4.35×10^5	2.9	1.90×10^5	4.0	1.03×10^5	4.4
0.5	2.45×10^5	3.7	1.07×10^5	4.6	5.84×10^4	6.6
1.0	1.57×10^6	2.9	6.85×10^5	2.9	3.74×10^5	2.8
1.0	1.19×10^6	3.0	5.22×10^5	2.6	2.84×10^5	2.8
1.0	8.15×10^5	3.2	3.56×10^5	3.0	1.94×10^5	3.1
1.0	4.35×10^5	3.5	1.90×10^5	3.2	1.03×10^5	4.7
1.0	2.45×10^5	4.3	1.07×10^5	4.2	5.84×10^4	8.4
2.0	1.57×10^6	3.0	6.85×10^5	3.0	3.74×10^5	2.9
2.0	1.19×10^6	2.9	5.22×10^5	3.4	2.84×10^5	2.6
2.0	8.15×10^5	3.0	3.56×10^5	3.6	1.94×10^5	4.5
2.0	4.35×10^5	3.1	1.90×10^5	4.2	1.03×10^5	4.6
2.0	2.45×10^5	3.8	1.07×10^5	6.4	5.84×10^4	8.6
<u>Sulphur</u>						
0.5	1.56×10^6	19.0	6.85×10^5	50.0		
0.5	1.19×10^6	28.0	5.22×10^5	48.0		
0.5	8.15×10^5	22.0	3.56×10^5	47.0		
0.5	4.35×10^5	22.0	1.90×10^5	44.0		
0.5	2.45×10^5	30.0	1.07×10^5	43.0		
1.0	1.56×10^6	19.0				
1.0	1.19×10^6	23.0				
1.0	8.15×10^5	28.0				

(Continued)

TABLE IV (Continued)

COMPARISON OF REYNOLDS NUMBER AND MEASURED
PARTICLE MASS MEAN DIAMETER

Tube Length (in.)	Reynolds Number and Particle Diameter for Stated Tube Diameter					
	0.1875 in.		0.0820 in.		0.0446 in.	
	$\frac{DG}{\mu}$	$\frac{D}{p}$	$\frac{DG}{\mu}$	$\frac{D}{p}$	$\frac{DG}{\mu}$	$\frac{D}{p}$
		(μ)		(μ)		(μ)
Sulphur, (Cont'd)						
1.0	4.35×10^5	26.0				
1.0	2.45×10^5	30.0				
2.0					3.74×10^5	57.0
2.0					2.84×10^5	49.0
2.0					1.94×10^5	46.0
2.0					1.03×10^5	29.0
2.0					5.84×10^4	26.0
8.7 μ <i>Serratia marcescens</i>						
	0.070 in.		0.042 in.		0.020 in.	
	$\frac{DG}{\mu}$	$\frac{D}{p}$	$\frac{DG}{\mu}$	$\frac{D}{p}$	$\frac{DG}{\mu}$	$\frac{D}{p}$
		(μ)		(μ)		(μ)
1.0	5.85×10^5	11.5	3.51×10^5	11.1	1.67×10^5	11.0
1.0	4.45×10^5	11.6	2.67×10^5	12.0	1.27×10^5	11.2
1.0	3.04×10^5	11.7	1.82×10^5	12.3	8.66×10^4	11.4
1.0	1.62×10^5	12.5	9.70×10^4	13.3	4.63×10^4	12.4
1.0	1.06×10^5	14.5	6.34×10^4	13.5	3.02×10^4	14.0
2.0	5.85×10^5	11.4	3.51×10^5	11.0	1.67×10^5	9.0
2.0	4.45×10^5	11.1	2.67×10^5	11.6	1.27×10^5	9.2
2.0	3.04×10^5	11.6	1.82×10^5	12.4	8.66×10^4	9.9
2.0	1.62×10^5	12.7	9.70×10^4	12.8	4.63×10^4	10.5
2.0	1.06×10^5	13.1	6.34×10^4	13.3	3.02×10^4	11.3
3.0	5.85×10^5	10.5	3.51×10^5	10.0	1.67×10^5	8.9
3.0	4.45×10^5	10.8	2.67×10^5	10.2	1.27×10^5	9.0
3.0	3.04×10^5	10.9	1.82×10^5	10.4	8.66×10^4	9.8
3.0	1.62×10^5	11.1	9.70×10^4	11.1	4.63×10^4	10.6
3.0	1.06×10^5	11.6	6.34×10^4	11.8	3.02×10^4	11.1

side with one block resting on ball bearings. A trough was provided in the top of the steel blocks to contain a layer of the material to be tested. These blocks are shown in Figure 4.

The first device incorporating these steel blocks consisted of a table which could be tilted about a horizontal axis. One of the blocks was made stationary to the table while the other one was allowed to roll free on ball bearings. With the blocks adjacent, a layer of the material to be tested was placed on their upper surfaces, and the angle of tilt required to separate the blocks was measured. However, this apparatus was not sufficiently sensitive because of the very small resisting forces within the powder.

Another design, incorporating the steel blocks, used a micro-manipulator mechanism and a small suspended weight to apply the force necessary to separate the blocks and rupture the powder. Bearing grooves were highly polished and precision-grade ball bearings were obtained in an effort to obtain free rolling. However, the magnitude of the combined errors resulting from insensitivity of the manipulator and bearing friction was still greater than the magnitude of the bulk tensile strength of the powders; this design was also abandoned.

The next apparatus incorporated the original steel, powdered-holding blocks with one block mounted at the lower end of an 18-inch pendulum. This design eliminated the ball bearings and, except for the knife edges on which the pendulum was mounted, was quite frictionless. Except for the effect of the weight of the powder which made repeated adjustments necessary, this was a satisfactory system.

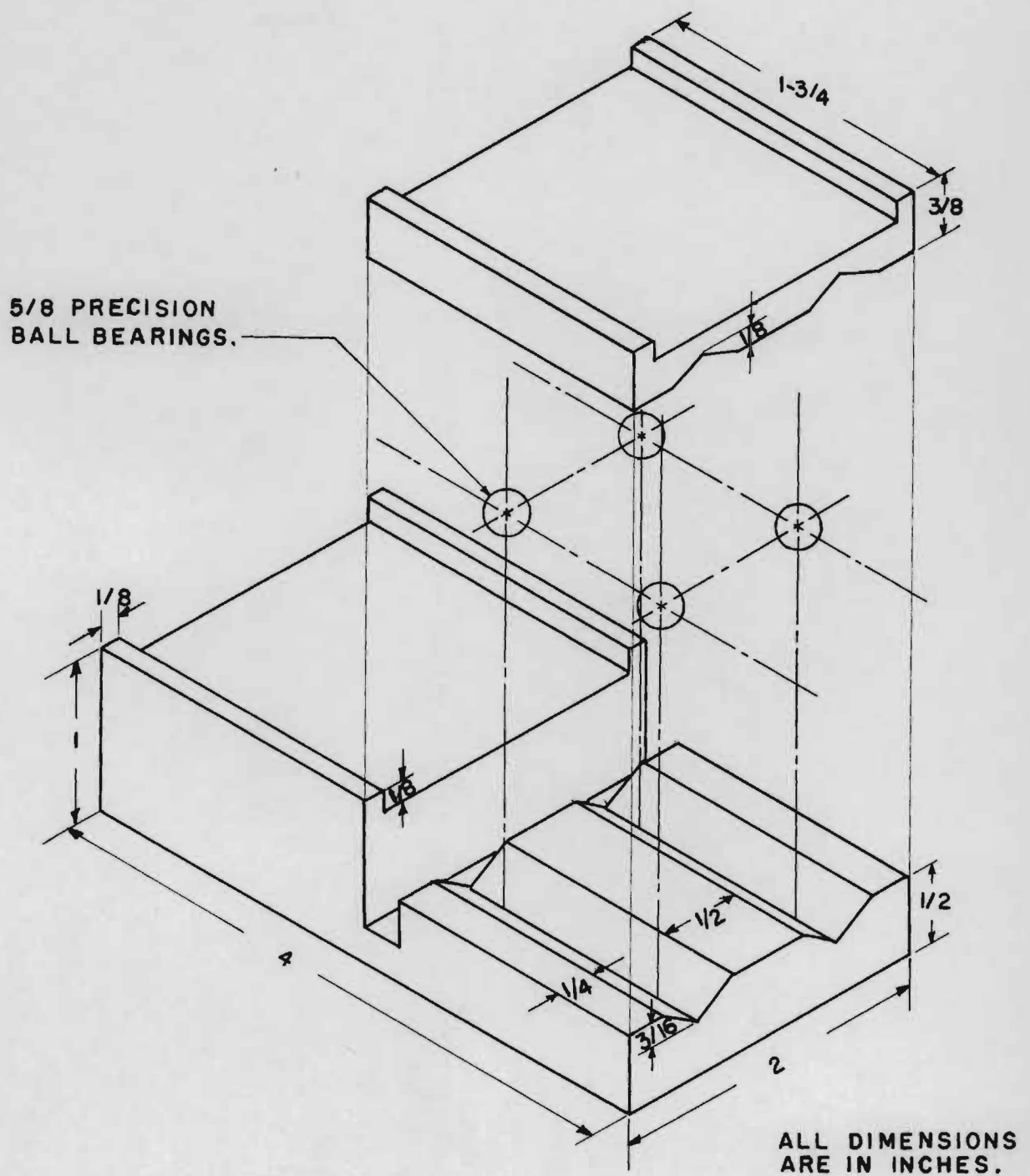


Figure 4. Sample Holder for Bulk Tensile Strength Apparatus.

The final and most generally useful design was actually two instruments in one. The force necessary to separate the blocks and to rupture the powder was produced with a torsion wire mechanism taken from a hydrophil balance manufactured by the Central Scientific Company, Chicago, Illionis. The magnitude of this applied force could be increased or decreased by changing the diameter of the torsion wire. The most satisfactory wire was a nichrome one of 0.020 in. diameter. Two separate bearing systems were used. For uncompacted or very lightly compacted powders one of the blocks holding the powder was suspended from a shaft vertically mounted on needle bearings. This arrangement is shown in Figure 5. For heavily compacted powders, ball bearings were used under the moving block as in previous designs in order to maintain a rigid platform on which to compact the powder. This arrangement is shown in Figure 6.

To ensure that the two blocks moved apart once the powder broke under the force exerted, a tiny permanent magnet was mounted so as to exert a small attraction on the part of the powder holder that moved, particularly when the two parts were together. Since magnetic field strength decreases rapidly with distance, this ensured a wide separation immediately after the sample of powder ruptured. The magnetic field was, of course, included in the zero calibration along with the force required to separate the blocks when there was no sample in the trough. In some of the early investigations with this device, a high resistance meter was used to indicate the breaking of contact, as shown in Figure 5. However, even the small currents involved seemed to weld the points of contact and to influence the breaking force slightly; use of the meter was discontinued.



Figure 5. Apparatus for Determining the Bulk Tensile Strength of Uncompacted Powders.

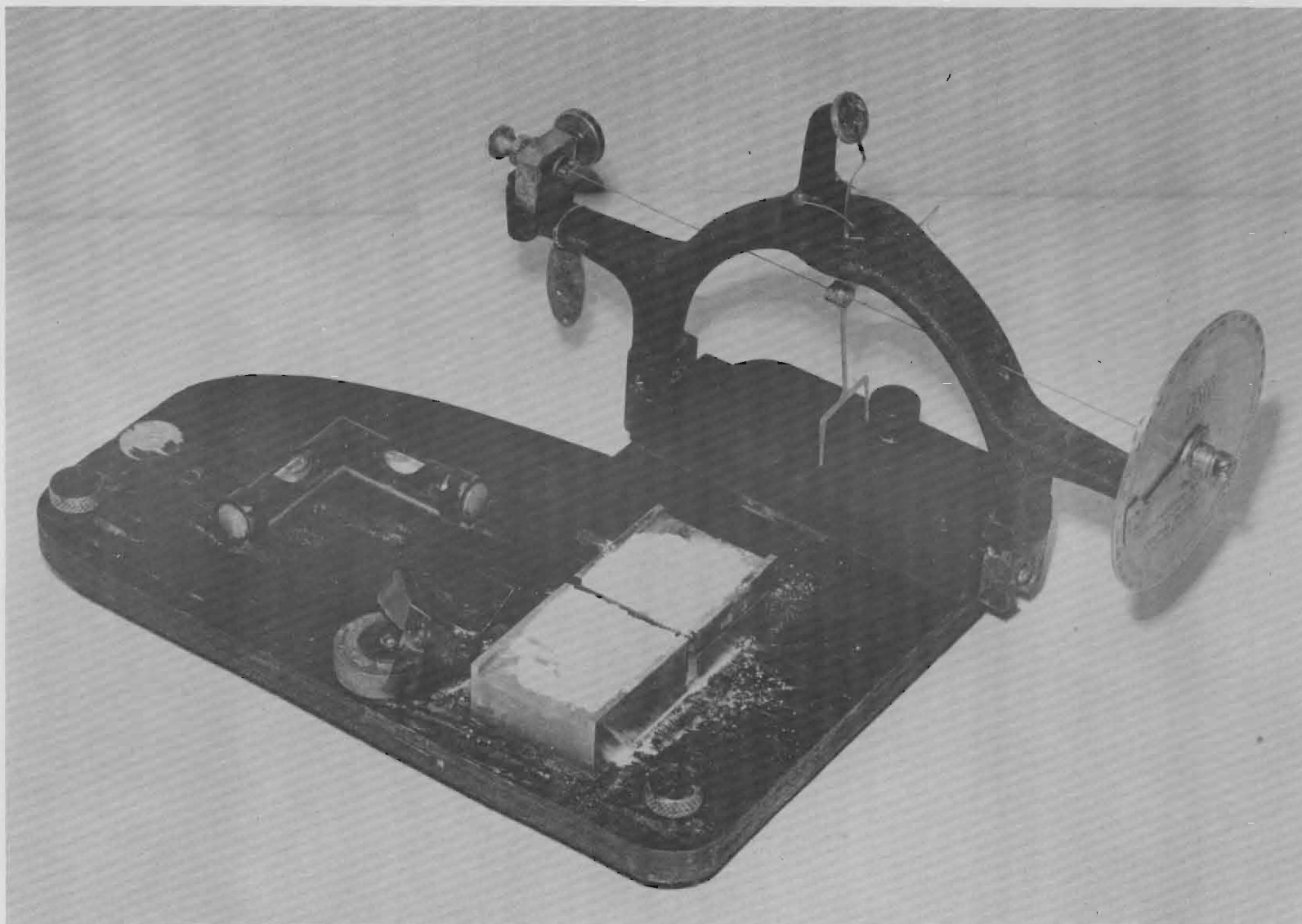


Figure 6. Apparatus for Determining the Bulk Tensile Strength of Compacted Powders.

Initially, bulk tensile strength measurements were made on powder samples that were dusted onto the trough and leveled off. These results were erratic and inconsistent, apparently because the loading of uncompacted samples could not be closely reproduced. In view of these results it was decided that more consistent data probably could be obtained by measuring the tensile strength of powders after they had been subjected to various applied compaction loads. This proved to be the case; extrapolation of the results to zero applied compaction load was taken as the uncompacted result.

The samples to be tested were prepared by spreading a layer of the material in the trough provided on the top of the steel blocks, which gave a layer of material approximately 2-1/2 inches long x 1-3/4 inches wide x 1/8 inch thick. A tetrafluorethylene block with a cross-sectional area of exactly 2 square inches was placed on this layer of material and additional weights were then placed on the plastic block. The weights and blocks were removed after a few minutes had elapsed and the tensile strength of the material determined by applying force with the torsion wire until the powder layer fractured. The depth of the compacted bed was then measured by means of a cathatometer and since the width was accurately known the cross-sectional area could be calculated. The samples tested were compacted under loads of 2.24, 1.68, 1.13, 0.58 and 0.25 lb/in².

Since the biological materials being investigated were extremely hygroscopic, it was necessary to conduct the tensile strength studies in a controlled atmosphere chamber. Such a chamber was constructed and is shown with the tensile strength apparatus in Figure 7. The tensile strength measurements for the biological materials were conducted in an atmosphere which was in

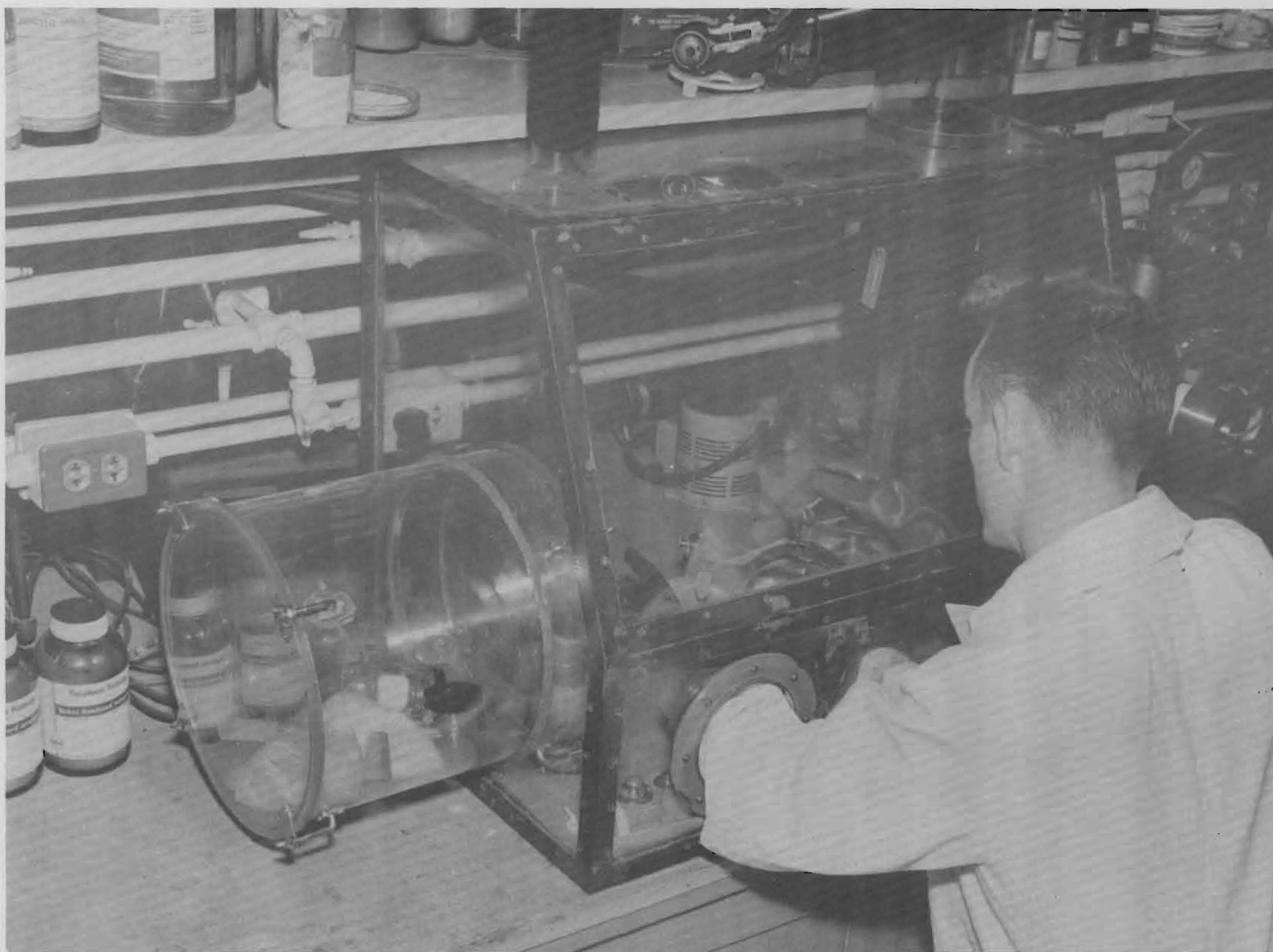


Figure 7. Dry Box Containing Bulk Tensile Strength Apparatus and Samples of Powders to be Tested.

equilibrium with a fresh surface of phosphorus pentoxide. Vapor pressure data for P_2O_5 at room temperature (approximately $25^{\circ}C$) indicated that the relative humidity of air in equilibrium with a fresh surface of P_2O_5 was less than one per cent. Though not accurate in this range, a hygrometer of Serdex, Incorporated, Boston, Massachusetts, was mounted in the chamber; it consistently indicated from 0 to 3 per cent relative humidity.

Both biological and inorganic powders were tested. At least five determinations were made on each powder at each compaction load; the average value was taken as the bulk tensile strength. These values and the percentage deviation are contained in Table XIV in the Appendix..

The average value of the bulk tensile strength for a particular compaction load was calculated by the equation

$$\frac{X_1 + X_2 + X_3 + \dots + X_n}{n} = a, \quad (25)$$

where X_1, X_2, \dots = individual tensile strength measurements,

n = number of measurements for a particular compaction load, and

a = average value of tensile strength for a particular compaction load.

The square of the standard deviation was next obtained by summing the squares of the differences between the individual values and the average value and dividing by the number of measurements in accordance with the equation

$$\frac{(X_1 - a)^2 + (X_2 - a)^2 + (X_3 - a)^2 + \dots + (X_n - a)^2}{n} = \sigma^2, \quad (26)$$

where σ = the standard deviation. The percentage deviation was finally obtained from the relationship

$$\frac{\sigma}{a} \times 100 = \text{percentage deviation.} \quad (27)$$

The average values for the bulk tensile strength of the materials tested, as given in Table XIV, were plotted and the best curve was drawn through the points. These curves (really straight lines) were extrapolated to zero applied compaction load to determine the bulk tensile strength of the powders when they were under no load other than their own weight. The bulk tensile strengths at zero applied compaction load are given in Table V.

C. Micromerograph Dispersion Studies

A series of investigations to indicate the relative dispersability of various biological materials supplied by the sponsor was undertaken. In these studies the Micromerograph with its usual deagglomerator was used. At the beginning of this series, rather wide ranges of pressures and deagglomerator settings were employed. The results indicated that adjusting the deagglomerator slit width at constant firing pressure was not as satisfactory as using various pressures at constant slit width. This was so because the large particle sizes of some of the powders prevented using anything but large deagglomerator settings. Thus, subsequent investigations were made using a slit width of 250 microns at firing pressures of 400, 300 and 200 psig. Results of these investigations are presented in Table VI. These data are the results of single runs, except in cases where the results of the first run appeared unusual. It should be borne in mind that these tests were not for the purpose

TABLE V

BULK TENSILE STRENGTH OF VARIOUS MATERIALS
FOR ZERO APPLIED COMPACTION LOADS

Material	Tensile Strength (lb/in. ² x 10 ³)	Tensile Strength (dynes/cm ²)	Relative Humidity (%)
<u>Bacillus subtilis</u> (acetone dried, washed, unground)	2.0	138	< 1 ⁺
<u>Bacillus subtilis</u> (Stokes dried, washed, unground)	2.2	152	< 1 ⁺
<u>Serratia marcescens</u> (3.3μ mmd)	2.3	159	< 1 ⁺
<u>Bacillus subtilis</u> (azeotropic dried, unwashed, unground)	2.7	186	< 1 ⁺
<u>Bacillus subtilis</u> (acetone dried, unwashed, unground)	3.8	262	< 1 ⁺
Cadmium Oxide (2.9μ mmd)	4.4	303	70
<u>Serratia marcescens</u> (freeze dried, unwashed, unground)	4.5	310	< 1 ⁺
<u>Serratia marcescens</u> (freeze dried, washed, unground)	4.7	324	< 1 ⁺
<u>Serratia marcescens</u> (8.7μ mmd)	5.7	393	< 1 ⁺
Zinc-Cadmium Sulfide (FP)	6.0	414	19
<u>Serratia marcescens</u> (3.9μ mmd)	6.3	434	< 1 ⁺
<u>Bacillus subtilis</u> (unground)	6.8	469	< 1 ⁺
Acrylic Resin (FT-3)	8.6	593	70
<u>Serratia marcescens</u> (12.6μ mmd)	8.8	607	< 1 ⁺

⁺ These determinations were made in an atmosphere which was in equilibrium with a fresh surface of P₂O₅ at approximately 25°C.

TABLE VI

DISPERSION OF BIOLOGICAL MATERIALS
BY THE MICROMEROGRAH DEAGGLOMERATOR

Pressure (psig)	Deagglomerator Opening (μ)	Cumulative Size Distribution								
		10%	20%	30%	40%	50%	60%	70%	80%	90%
		(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)
<u>Serratia marcescens</u> 242 (freeze dried, washed, ground)										
400	250	4.3	5.3	6.2	7.1	8.2	9.3	10.9	13.0	17.3
300	250	4.3	5.3	6.2	7.1	8.2	9.3	10.9	13.0	17.3
200	250	4.5	5.7	6.6	7.6	8.4	9.6	10.9	13.0	17.3
<u>Serratia marcescens</u> 242 (freeze dried, unwashed, ground)										
400	250	4.1	5.2	6.0	6.7	7.5	8.4	9.5	11.4	15.5
300	250	4.6	5.9	6.7	7.5	8.4	9.4	10.5	12.5	15.8
200	250	4.9	6.0	7.2	8.2	9.1	10.4	11.9	14.3	18.1
<u>Serratia marcescens</u> 242 (freeze dried, washed, unground)										
400	200	3.9	5.3	6.3	7.3	8.4	9.6	10.8	13.5	17.2
400	200	3.7	4.8	5.8	6.8	7.8	9.1	10.5	14.2	30.0
400	250	4.3	5.6	6.6	7.5	8.8	10.1	11.8	14.8	19.0
<u>Serratia marcescens</u> 242 (freeze dried, unwashed, unground)										
400	100	4.1	5.4	6.6	7.7	8.9	9.9	11.5	14.2	18.5
400	200	4.9	6.1	7.2	8.5	9.8	11.2	13.0	15.5	21.0
400	250	4.6	5.9	6.9	8.0	9.2	10.5	12.8	15.5	24.0
200	150	5.0	6.6	8.0	9.4	10.8	12.4	14.5	17.2	23.0
200	200	5.1	6.7	8.1	9.6	10.7	12.3	14.6	17.5	25.0
100	200	5.6	7.5	8.9	10.3	12.0	14.0	16.3	18.9	26.0
<u>Bacillus subtilis</u> (Stokes dried, unwashed, ground)										
400	250	4.5	6.5	8.2	10.0	12.5	15.2	18.0	22.4	37.0
300	250	4.5	6.1	7.5	9.1	10.5	13.0	16.0	19.5	32.0
200	250	6.1	8.1	10.2	13.6	16.0	19.0	23.0	32.0	48.0
<u>Bacillus subtilis</u> (Stokes dried, washed, unground)										
400	250	5.1	6.9	8.5	10.1	12.3	14.5	18.1	29.0	68.0
300	250	6.6	9.3	12.0	15.0	18.0	22.0	34.0	66.0	---
200	250	7.6	10.8	15.4	20.0	25.2	34.5	47.0	71.0	---
<u>Bacillus subtilis</u> (azeotropic dried, unwashed, ground)										
400	250	3.5	4.4	5.1	5.8	6.5	7.2	8.2	9.4	12.0
300	250	3.8	4.8	5.7	6.5	7.4	8.2	9.3	11.0	14.0
200	250	4.3	5.7	6.7	7.6	8.6	9.6	11.1	13.0	15.5

(Continued)

TABLE VI (Continued)

DISPERSION OF BIOLOGICAL MATERIALS
BY THE MICROMEROGRAH DEAGGLOMERATOR

Pressure (psig)	Deagglomerator Opening (μ)	Cumulative Size Distribution								
		10%	20%	30%	40%	50%	60%	70%	80%	90%
		(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)
<u>Bacillus subtilis</u> (azeotropic dried, unwashed, ground)										
400	250	3.5	4.4	5.1	5.7	6.4	7.3	8.3	9.5	12.0
300	250	3.6	4.5	5.2	6.0	6.8	7.6	8.7	10.0	14.5
200	250	3.6	4.6	5.4	6.4	7.3	8.3	9.5	11.1	14.6
<u>Bacillus subtilis</u> (azeotropic dried, unwashed, unground)										
400	150	3.2	4.3	5.3	6.5	7.7	9.3	11.8	16.0	25.0
300	150	4.2	5.4	6.5	7.5	8.6	9.7	11.2	13.5	17.0
200	150	3.0	4.0	5.1	5.9	6.7	7.8	8.8	9.8	11.0
100	150	3.9	5.5	7.0	8.4	9.4	10.8	12.8	15.1	19.3
50	150	3.6	5.5	7.0	8.4	9.4	10.8	12.8	14.6	17.0
400	250	4.4	5.7	6.8	7.9	9.0	10.1	12.0	14.5	18.5
400	250	4.4	5.7	6.7	7.9	9.1	10.1	12.1	14.7	19.0
400	150	4.9	6.1	7.5	8.7	10.1	10.8	13.6	16.5	21.0
400	150	4.3	5.6	6.6	7.7	8.9	10.2	11.8	14.5	19.0
400	100	4.2	5.6	6.6	7.8	9.0	10.2	11.8	14.5	19.0
400	75	4.4	5.8	7.0	8.2	9.5	10.8	13.0	16.0	21.5
400	50	4.3	5.7	6.8	8.0	9.3	10.5	12.2	15.2	20.2
<u>Bacillus subtilis</u> (acetone dried, washed, ground)										
400	250	3.0	3.8	4.7	5.6	6.4	7.2	8.4	10.0	13.0
300	250	2.8	3.6	4.4	5.5	6.4	7.4	8.9	10.4	14.5
200	250	3.2	4.2	5.2	6.4	7.4	8.6	10.1	12.0	15.5
<u>Bacillus subtilis</u> (acetone dried, unwashed, ground)										
400	250	3.2	3.9	4.5	5.1	5.7	6.5	7.4	8.7	10.7
300	250	3.1	3.6	4.2	4.9	5.6	6.4	7.4	8.7	10.7
200	250	3.6	4.6	5.5	6.3	7.0	8.2	9.4	10.6	14.0
<u>Bacillus subtilis</u> (acetone dried, washed, unground)										
400	250	2.9	3.6	4.3	5.4	6.5	7.6	9.3	13.0	40.0
300	250	3.4	4.3	5.5	6.8	7.9	9.4	11.5	16.0	55.0
200	200	3.5	4.9	6.1	7.7	9.3	10.9	13.6	19.0	65.0
<u>Bacillus subtilis</u> (acetone dried, unwashed, unground)										
400	200	3.8	5.2	6.3	7.3	8.7	10.0	12.6	19.0	50.0
200	150	4.7	6.1	7.6	9.2	10.5	12.0	15.0	19.2	33.0
200	200	4.2	5.8	7.3	8.8	10.6	12.6	16.5	26.0	60.0
200	250	4.2	5.8	7.3	8.8	10.6	13.6	19.0	35.0	70.0

(Continued)

TABLE VI (Concluded)

DISPERSION OF BIOLOGICAL MATERIALS
BY THE MICROMEROGRAH DEAGGLOMERATOR

Pressure (psig)	Deagglomerator Opening (μ)	Cumulative Size Distribution								
		10%	20%	30%	40%	50%	60%	70%	80%	90%
		(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)
Zinc-Cadmium Sulfide (FP)										
400	250	2.6	3.6	4.6	5.7	7.0	8.2	9.7	11.3	13.8
200	250	4.6	6.1	7.5	8.8	10.0	11.5	13.2	15.5	18.2
Acrylic Resin (FT-3)										
400	250	3.6	4.5	5.3	6.0	6.6	7.4	8.4	9.7	11.5
200	250	4.6	5.7	6.6	7.4	8.2	9.0	10.2	11.5	13.4
Cadmium Oxide (2.9 μ mmd)										
400	250	1.3	1.8	2.2	2.5	2.9	3.4	3.8	4.4	5.2
200	250	1.4	1.8	2.2	2.5	2.9	3.4	3.9	4.6	5.5

of obtaining the ultimate size distribution of the powders but were intended to show the effects of various shears.

D. Auxiliary Studies

1. Shear in Powders

a. Early Attempts. Following the technique outlined of Cremer, Conrad and Kraus⁵, a device was set up consisting simply of a flat, metal plate which could be rotated slowly about a horizontal axis. The angle through which rotation had occurred could be read from an indexed scale with vernier. With such a device a measure of the "adhesiveness" of powders was sought.

5. Cremer, E., Conrad, F., and Kraus, Th., "Die Haftfähigkeit Von Pulvern und ihre Anwendung Zur Bestimmung Von Korngroszen," Angew Chem. 64, No. 1, 10-11 (1952).

The experimental procedure for obtaining powder shear information was, first, to establish a film of particles adhering to the flat plate. This was done by spreading a thin film of adhesive on the plate, and then sifting a rather thick layer of particles onto the adhesive. When dry, the excess particles and those not firmly attached were blown off. Another layer of particles was then screened onto the adhering layer. In the technique first employed, the plate was rotated until all of, or more usually a portion of, the upper particle layer slid off. The weight of the sliding material was then obtained, and, using the analysis of Gremer, Conrad and Kraus, the data were plotted with $mg \sin \phi$ as the ordinate and $mg \cos \phi$ as the abscissa. In this system, m is the mass of displaced powder, g is the acceleration of gravity and ϕ is the angle through which the plate was turned from the horizontal. The result to be expected, according to the original investigators, was a straight line having a positive intercept on the ordinate. In general, this was the case in the present work, but there was a rather large spread to the data. Using sized fractions of magnesite and iron powder having diameters between 300 and 60 μ Gremer, et al., obtained data indicating a rather straight line increase in adhesive force with particle size decrease.

In an effort to improve the technique, particles were established on the flat, metal plate as before. Other plates, having areas of 0.75 in.² to which also had been attached a layer of particles with adhesive, were placed, with the particles down, on the first plate. When rotated, shear thus had to occur in the unattached layer of particles trapped between the two plates. The weight of the upper plate with its attached particles determined the mass causing the shear. Six plates of different weights were used. The data, treated as before, indicated little, if any, improvement in reproducibility.

b. Rotating Apparatus

(1) Description. Rather than depend on gravity to produce the shearing force and in order to utilize a larger area of shear, a rotary test was arranged to determine the shear strength of the powdered materials. The apparatus consisted of a motor-driven turntable mounted on precision bearings. The rate of rotation of the motor, and hence of the turntable, could be controlled through a voltage regulator attached to the motor. A special sample holder was mounted on the turntable. This holder consisted of a metal base plate, a clear plastic cover and separate disks on which to prepare the test samples. The disks and samples fitted into a recessed area on the base plate. The plastic holder, when clamped onto the base plate, provided an air-tight seal and prevented air currents from disturbing the sample during rotation.

(2) Procedure. The samples were prepared on the circular disks as before, i.e., a thin layer of adhering particles was covered with another layer. When placed in the apparatus, the plastic cover was attached, and the turntable was started. Its rate of rotation was determined with a Strobotac of the General Radio Company, Cambridge, Massachusetts. As rotation increased, a free zone around the periphery was first produced and then progressed toward the center as the unattached particles were slung off. Periodically the apparatus was stopped. The area of remaining particles was estimated, and the weight of the particles removed was obtained.

(3) Calculations and Results. Figure 8 is a schematic representation of the results of an experimental test.

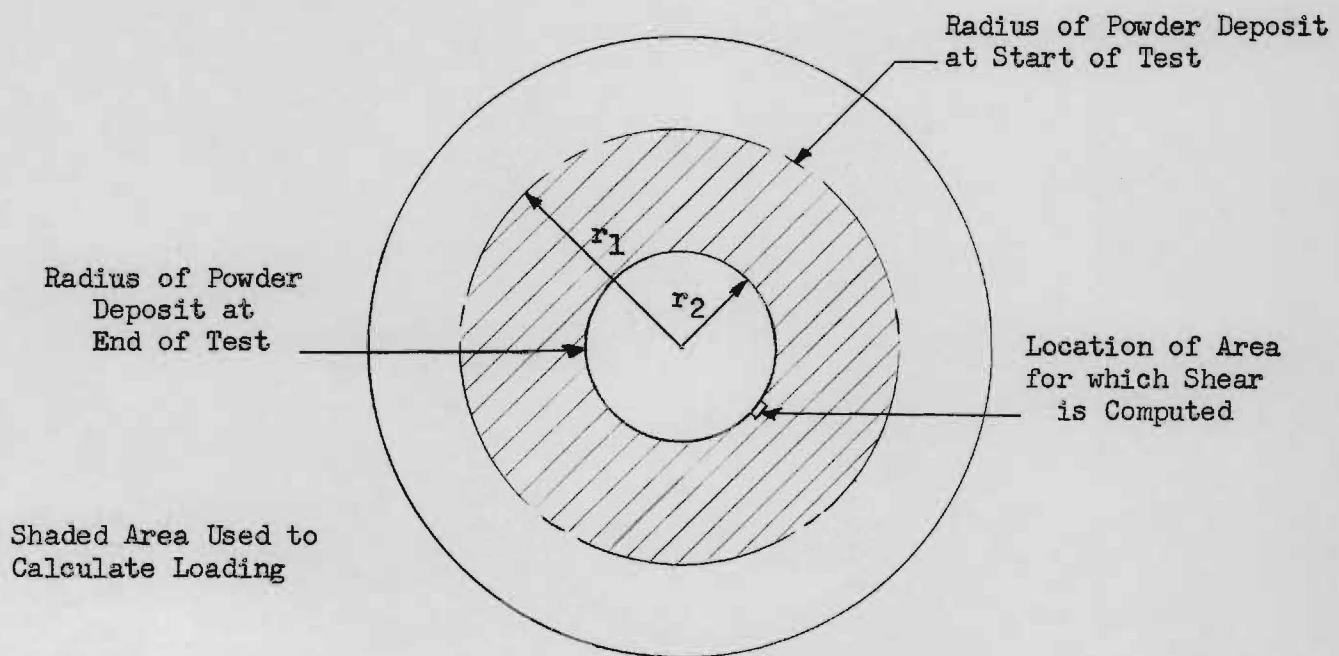


Figure 8. Schematic Diagram of Centrifugal Shear Experiment.

The loading of powder on the disk was calculated by

$$W = \frac{\text{weight loss per test}}{\pi (r_1^2 - r_2^2)}, \quad (28)$$

where the weight loss per run is the initial weight of the disk at radius r_1 minus the final weight at radius r_2 in grams; r_1 is the initial radius of powder in inches, and r_2 is the final radius of powder in inches. The shearing force per unit area, based on the final radius, was calculated by

$$S = k \cdot W \cdot r_2 \cdot \omega^2, \quad (29)$$

where S = shearing force per unit area, dynes/mm²,
 k = a conversion factor, 4.34×10^{-5} ,
 G = sample loading, g/in.²,
 r_2 = final radius of remaining powder, and
 ω = angular velocity, rpm.

Tables XV, XVI, XVII, XVIII, XIX, and XX in the Appendix give the calculated and experimental data for some of the powders investigated. The adherence of most powders was so low that the method gave quite inconsistent results. The tests were stopped in favor of other systems.

2. Humidity Effect Studies

A series of investigations to determine the effect of relative humidity on forces of adhesion was conducted, using an apparatus assembled around a Chainomatic Analytical Balance (Christian Becker, Incorporated, New York) with the pans removed. A glass sphere approximately 5 mm in diameter was suspended by means of a nylon monofilament from one balance arm, directly above a second sphere resting on a small stand. Auxiliary equipment included a small radioactive source to minimize electrostatic charging, a Laboratory Standard Hygrometer (Serdex, Incorporated, Boston, Massachusetts), a heat lamp to dry the spheres, and a microscope located outside the balance case for viewing the spheres.

Tests were conducted by first adjusting the balance arm with the suspended sphere to zero. The spheres were then brought together, and the force required for separation was determined. In some cases, a visible amount of water was present between the spheres; in others, the water was evaporated before separation. In other cases, no visible amount of water was present.

At normal room humidities, the forces required to separate the spheres appeared to be so small as to be nearly immeasurable with the system employed, if the spheres were in equilibrium with the water vapor in the air when brought together. When non-equilibrium moisture (i.e., water drops placed on the spheres by means of a hypodermic syringe which were not in equilibrium with the water vapor in the surrounding air) was present, the forces required to separate the spheres were found to increase with a decrease in the amount of water between them. However, when the spheres are brought together wet, and then dried before separation, the forces required are considerably greater than those found with non-equilibrium water present. These investigations were of an exploratory and qualitative nature. Time and other work which were deemed more pressing did not permit full exploration.

3. Centrifuge Studies

The data contained in the semifinal report⁶ for glass beads adhering to a plastic surface indicate that a linear relationship exists between the rate of rotation and the inverse of the diameter of the largest particles adhering. This diameter was taken as the particle the diameter of which was larger than 98 per cent of those remaining. The actual relationship between rate of rotation and particle diameter is of the form,

$$r = K/D_p, \quad (30)$$

where r = revolutions/second,

D_p = diameter of particle, and

K = constant.

⁶. Orr, et al., op. cit., p. 89.

Substitution of equation 30 into the centrifugal force equation,

$$F = \rho \times 1/6 \pi D_p^3 \times (2\pi r)^2 R, \quad (31)$$

where F = force exerted on the particle,

ρ = density of particle,

D_p = diameter of particle,

r = revolutions/second, and

R = distance from center of rotor to particle location within
the cell,

shows that the force of adhesion is directly proportional to the particle diameter.

Similar tests were also conducted with glass beads on a glass surface. This time the number of glass spheres and their size distribution were obtained. In the first series of tests, the data of which are contained in TableXXI in the Appendix, the samples of spheres chosen had such a small average size that little change in the distribution was produced. The second series was more successful in that a greater change in the distribution was obtained, but the distribution was not a normal one. These data are given in TableXXII in the Appendix. For the above reasons these latter data could not be analyzed to determine if a relationship, similar to the one for glass beads on a plastic plate, existed for glass beads on a glass plate.

IV. DISCUSSION OF RESULTS

A. Capillary Deagglomeration

The analyses given in Section III, A-2, "Dispersion Analysis", are necessarily concerned with rather idealized situations, thus their applicability to practical situations is limited. Dimensional analysis, for example, requires that all of the variables governing the experiment be known and measurable. Further, to achieve a correlation of assured value, it is desirable that each of the dimensionless groups be varied independently. This form of analysis, therefore, finds its greatest usefulness in the design of experiments. However, despite popular misconceptions, it does not provide a sure method for obtaining a correlation of experimental data, nor does it impose any limitation whatsoever on the form of the correlation function. The analysis is, in general, applicable to any system for which the assumptions made in the derivations are valid. The method involves no empirical approximations; however, to apply it to the experimental work necessitates the use of empirical methods to determine the magnitude of several of the variables involved.

Though an unsteady-state flow system was employed, the powders emerged from the capillary tubes in such a short time interval that pressures and flow rates changed little. Conditions for the powder, therefore, approximated steady-state. Attempted correlations of data assumed constancy of conditions.

Correlation of the three dimensionless groups $-- \frac{DG}{\mu}$, $\frac{D_p}{D}$ and $\frac{L}{D} --$ in a function of the form

$$\frac{D_p}{D} = \alpha \left(\frac{DG}{\mu} \right)^a \left(\frac{L}{D} \right)^b \quad (32)$$

for the materials investigated was not satisfactorily attained. Though individual materials could be correlated by selected values of α , a and b , no single correlation for all materials was found. The results are deemed most informative as presented in Table IV.

Since the Reynolds number is a criterion for dynamic similitude and is also a measure of the ratio of inertial to viscous forces in the dispersing gas, this dimensionless group was selected as a basis for comparing the degree of deagglomeration obtained. From Table IV, it can be seen that, in general, the mass mean diameter for a particular tube length and diameter increases with decreasing Reynolds number. There is a strong indication in the results of the most extensive investigation--the 8.7μ Serratia marcescens--that increasing tube length at constant Reynolds number and diameter increases the deagglomeration; however, this effect is not as pronounced as in the decreasing Reynolds number. Decreasing the tube diameters, in general, produces better deagglomeration.

It has been previously suggested⁷ that the deagglomeration produced by small tubes is possibly due to the shear at the wall as described by the modified form of the Blasius shear equation,

$$T_w = \frac{d \Delta p}{4L} \quad (33)$$

7. Orr, et al., op. cit., p. 173.

where T_w = shear at the wall,

Δp = pressure drop across the tube,

d = diameter of the tube, and

L = length of the tube.

Further attempts at correlating this maximum shear with the degrees of deagglomeration achieved have shown that this does not adequately describe the dispersion process. This is probably due to the fact that the short tube lengths investigated do not allow all the particles to pass through this shear zone before they pass from the tube. Time did not permit investigation of the tube lengths required to determine if the shear at the wall becomes a major factor in the dispersion process.

An attempt to determine the effect of the Micromerograph channel on the mass mean diameter of the powders dispersed through the tubes was made for the 8.7μ Serratia marcescens and also the unground Bacillus subtilis. These investigations were conducted to determine the particle size of the powder produced on passage through the Micromerograph channel at Reynolds numbers equivalent to those with the tubes in place. The results indicated that the degree of deagglomeration produced by the channel alone is negligible compared to that produced by the tube. This is shown by a comparison of the data of Table XII and Table XIII. In fact, it was sometimes extremely difficult to produce Reynolds numbers in the channel alone that correspond to the Reynolds number in the channel with tube attached.

For example, the highest Reynolds number attained in an 0.020 in. diameter tube was 167,000 and the lowest was 26,100, a range of 6.4 to 1. The Micromerograph channel diameter was greater than that of the tube by

a factor of about 9. Thus when the highest Reynolds number was reached in the tube, that in the channel was below the minimum number attained in the tube.

B. Bulk Tensile Strength of Powders

From the data of Table V, it can be seen that no general correlation of particle size and bulk tensile strength is evident. Cremer, Conrad and Kraus⁸ found that for the materials they tested the adhesive strength increased with decreasing particle size. However, these investigators were using inorganic materials in narrow particle size ranges. It is most probable that for the biological materials considered in this report such factors as particle size ranges covering one order of magnitude, the presence of foreign, inhomogeneous matter, (i.e., the nutrient in which the culture was produced) electrostatic charging, moisture sensitivity even at low relative humidities, and the presence of large aggregates of particles obscured any relationship that might exist between the bulk tensile strength and some other property of the powders. Electrostatic charging was a particularly noticeable factor for some of these materials, especially the 12.6 μ mmd sample of Serratia marcescens. This charging effect was characterised by the powders 'leaping' from one surface to another; particularly plastic surfaces. Electrostatic charging produced higher tensile strength values for the powders exhibiting this effect than those that did not.

From the data of Table V, it appears, in general, that for a particular material the washed sample has a lower bulk tensile strength than the

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8. Cremer, et al., op cit., p. 10-11.

unwashed sample for any particular method of drying.

C. Micromerograph Dispersion Studies

The results of the dispersion studies using biological materials indicate that a general comparison on the basis of the types of treatment can be made. A measure of dispersibility was arbitrarily taken to be the mass mean diameter obtained with a dispersing pressure of 200 psig divided by the lowest mass mean diameter obtained (generally the value for the 400-psig test). Thus, the nearer unity of the value the more easily is the powder dispersed. Using this measure as a guide, the types of powders were ranked in order of decreasing dispersibility (increasing difficulty for dispersion); these data are contained in Table VII. Using these data as a guide, the types of powders ranked in order of ease of dispersion are: freeze dried Serratia marcescens, acetone dried Bacillus subtilis, azeotropic dried Bacillus subtilis, and Stokes dried Bacillus subtilis. The washed materials, in general, were found to be more readily dispersed than the unwashed.

From Table V, containing data on bulk tensile strengths of the powders, and Table VII it can be seen that the powders with the lowest dispersibility factor do not necessarily have the lowest bulk tensile strength. At first, this seems a discrepancy in the data. In reality, these two sets of data are probably measurements of two different properties. The powders considered here have, in their bulk form, many small lumps or tight aggregates of powder surrounded by relatively loose unaggregated powder. When the bulk tensile strength is measured, as outlined in this report, these aggregates are not broken; the loose unaggregated powder around the lumps separates. On the other hand, when the powder is dispersed in the deagglomerator it is the

TABLE VII
DISPERSIBILITY FACTORS FOR VARIOUS MATERIALS

Material	Dispersibility Factor
Cadmium Oxide (2.9 μ mmd)	1.00
<u>Serratia marcescens</u> 242 (freeze dried, washed, ground)	1.03
<u>Serratia marcescens</u> 242 (freeze dried, unwashed, unground)	1.09
<u>Bacillus subtilis</u> (azeotropic dried, unwashed, ground)	1.14
<u>Bacillus subtilis</u> (acetone dried, washed, ground)	1.15
<u>Serratia marcescens</u> 242 (freeze dried, unwashed, ground)	1.21
<u>Bacillus subtilis</u> (acetone dried, unwashed, unground)	1.22
Acrylic Resin (FT-3)	1.24
<u>Bacillus subtilis</u> (acetone dried, unwashed, ground)	1.25
<u>Bacillus subtilis</u> (Stokes dried, washed, ground)	1.28
<u>Bacillus subtilis</u> (azeotropic dried, washed, ground)	1.32
Zinc-Cadmium Sulfide (FP)	1.43
<u>Bacillus subtilis</u> (acetone dried, washed, unground)	1.43
<u>Bacillus subtilis</u> (Stokes dried, washed, unground)	2.05

breakup of these tight aggregates that is of importance. Therefore, since two different mechanisms likely are involved, these two measures, the bulk tensile strength and the measure of dispersibility, may not be directly related. This physical picture of the state of the powder is in agreement with a similar conclusion by Carman⁹.

9. Carman, P. C., "The Surface Area Measurement of Paint Pigments." J. Oil and Colour Chemists Assoc., p. 165, April (1954).

D. Auxiliary Studies

1. Shear in Powders

Early attempts to measure the shear required to move one layer of particles over another using a gravitational device were, in general, unsuccessful. The major difficulty in this series of investigations was the lack of reproducibility; this effect is probably due to variations in the powder layers. This method was subsequently abandoned, and investigations using the rotary device were begun. Here again, as can be seen in Tables XV, XVI, XVII, XVIII, XIX, and XX, considerable difficulty was encountered in obtaining reproducibility. The major error introduced here was the determination of the radius of the sheared powder mass; it was also noted that many materials sheared at a low rate of rotation, thus preventing an accurate measurement of the shear.

2. Humidity Effect Studies

The series of investigations to determine the force of adhesion between two glass spheres at various relative humidities was not conducted beyond the preliminary stage. The indications from the preliminary work are that this method would be quite satisfactory; these studies were abandoned, however, so that more tests with biological materials could be conducted.

3. Centrifuge Studies

The investigations to determine the size distribution of glass beads remaining on a solid surface after subjection to increasing centrifugal force fields were rather inconclusive. Only a few investigations using this method were made; however, the general trend indicates that the larger spheres tend to be removed first, with only a few small spheres being removed at

low rates of rotation. This series of investigations was also terminated to permit more emphasis on biological materials of direct interest to the sponsor.

V. CONCLUSIONS

From the results of this investigation a number of definite conclusions and general observations can be drawn. Among the more definite conclusions are the following:

1. With capillary tubes as the dispersing mechanism, increasing deagglomeration results from higher gas pressure, increased tube length, decreased tube diameter and, for each tube, increased Reynolds number.

2. Essentially complete deagglomeration (separation of particles but not particle fracture) was attained for all powders, including bacterial powders. For the finest powders this required a gas pressure of 400 lb/in.^2 , a tube diameter no greater than 0.02 in. and a tube length of as much as 3 in.

3. The tensile strengths of bulk, dry, biological powders are in the range of 2×10^{-3} to $9 \times 10^{-3} \text{ lb/in.}^2$, or considerably lower than is thought to be the actual strength of the material composing the powder.

4. The tensile strengths of bulk, uncompacted "FT-3" and "FP" were 8.6×10^{-3} and $6.0 \times 10^{-3} \text{ lb/in.}^2$, respectively.

5. There appears to be no definite correlation between the bulk tensile strength of a powder and its ease of dispersibility probably because these are measurements of two different properties. It is probable the powders considered here are composed, in their bulk form, of many small lumps or tight aggregates of powder surrounded by relatively loose unaggregated powder. When the bulk tensile strength is measured, as outlined in this report, these aggregates are not broken; it is the loose, unaggregated powder around the lumps that separates. On the other hand, it is the break-

up of the tight aggregates that is of importance when the powder is dispersed in the deagglomerator.

6. The dispersion techniques and the tensile strength apparatus as developed for this work are recommended for future work requiring such data. A method for determining the actual tensile strength between two individual particles would be a valuable aid in studies of this type.

7. The force of adhesion of spheres to solid surfaces, under rather ideal conditions, was found to be directly proportional to the particle diameter.

Observations of a general nature are as follows:

1. Water vapor has a definite influence on the cohesiveness and dispersibility of powders, but just what is the influence was not determined. The relationship does not appear to be a simple one, e.g., it probably alters adhesive and electrostatic effects in many cases. Bacterial materials were handled in dry atmospheres (less than 1 per cent relative humidity).

2. The tube-dispersion data would be expected to give, at least, an engineering correlation involving some combination of viscous, inertial and tensile forces. Because most of the materials investigated were quite similar, it was not possible to vary greatly some of the properties; this probably prevented achievement of a correlation, since the data, particularly that of later phases of the work, seem to be quite consistent.

3. The powder being analyzed in a Micromerograph passes the deagglomerator with the first quantities of propelling gas. Dispersion can, therefore, be said to have been carried out at essentially the initial gas pressure.

VI. RECOMMENDATIONS

Liquid atomization, having been investigated much more thoroughly over a number of years, is now reasonably predictable. The same will undoubtedly be accomplished for powder deagglomeration in time. The approach used here, viz., actual tensile strength measurements (which include any effects due to moisture, electrification, and the like) coupled with actual deagglomeration tests, is thought to be a good start. The time for inclusion of specific powder characteristics will come after a basic correlation is attained.

Specifically, further work of the nature outlined is recommended as the approach to lead most directly to predictable powder dispersion. For general usefulness, powders having a wide variety of densities, tensile strengths, etc., should be included. To establish the effect of other conditions--preparation, aging, relative humidity, to name a few--on a single type of powder, dispersion with one fixed deagglomerator is recommended.

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VII. APPENDIX

SAMPLE CALCULATION OF FINAL TANK PRESSURE
AFTER COMPLETE POWDER DISPERSION THROUGH TUBES

The experiments using the motion picture technique to determine the length of time for completely dispersing the powder indicated that for all cases the time required for powder sample passage through the tubes was considerably less than one second. The test giving the longest time for dispersion yielded the following data:

$$\theta = 0.05 \text{ second}$$

$$D = 0.020 \text{ inch}$$

$$L = 3.0 \text{ inches}$$

$$\text{initial pressure} = 200 \text{ psig}$$

The final pressure can be calculated from equation 23:

$$\theta = \frac{2 N_o T_o^{1/2} \left(P_o^{\frac{1-k}{2k}} - P_o^{\frac{1-k}{2k}} \right)}{C (k-1) P_o^{\frac{k+1}{2k}}}$$

The constants in the above equation have the following values or meaning:

$$T_o = 530^\circ R,$$

$$k = 1.4,$$

$$C = \left(\frac{G}{G_{cni}} \right)^A \left(\frac{454}{M} \right) \left(\frac{g_c M}{e R} \right)^{1/2},$$

$$\frac{G}{G_{cni}} = 0.4,$$

$$M = 28,$$

$$g_c = 32.2,$$

$$e = 2.718, \text{ and}$$

$$R = 1546.$$

Writing the perfect gas law,

$$N_o = \frac{P_o V}{R T_o} = \frac{P_o \times 26.1}{82.04 \times 294 \times 144 \times 14.7}$$

$$N_o = 5.11 \times 10^{-7} P_o.$$

Substituting N_o into equation 23, and simplifying, gives

$$\theta = \frac{10.22 \times 10^{-7} T_o^{1/2}}{C (k - 1)} \left[\left(\frac{P_o}{P} \right)^{\frac{k-1}{2k}} - 1 \right].$$

Calculating the value of C:

$$C = 0.4 \times \frac{\pi}{4} \times \frac{(0.020)^2}{144} \times \frac{454}{28} \left(\frac{32.2 \times 28}{2.718 \times 1546} \right)^{1/2}$$

$$C = 6.56 \times 10^{-6}.$$

Substituting into the above equation gives:

$$0.05 = \frac{1.022 \times 10^{-6} \times 23}{6.56 \times 10^{-6} \times 0.4} \left[\left(\frac{P_o}{P} \right)^{0.143} - 1 \right]$$

$$\left(\frac{P_o}{P} \right)^{0.143} = 1 + \frac{0.05 \times 0.4 \times 6.56}{1.022 \times 23}$$

$$\left(\frac{P_o}{P}\right)^{0.143} = 1 + 0.00558 = 1.00558$$

$$P^{0.143} = \frac{P_o^{0.143}}{1.00558} = \frac{(214.7)^{0.143}}{1.00558} = 2.138$$

$$P = 205 \quad \text{psia} = 190 \text{ psig.}$$

Thus the pressure in the system when the last bit of powder emerged was only about 10 lb/in.² less than when the powder started through the tube.

TABLE VIII

DEAGGLOMERATION OF 3.3 μ *Serratia marcescens* BY SMALL TUBES

Deagglom- eration Pressure (lb/in. ²)	Tube Diameter (in.)	Cumulative Size-Frequency Distribution								
		10%	20%	30%	40%	50%	60%	70%	80%	90%
		(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)
1/2-in. tube										
400	0.1875	2.7	3.4	4.1	4.8	5.4	5.9	6.8	7.9	9.3
400	0.082	2.3	2.7	3.1	3.4	3.7	3.9	4.1	4.5	5.0
400	0.0446	1.9	2.4	2.9	3.4	3.9	4.5	5.2	6.3	8.0
300	0.1875	3.0	3.8	4.5	5.1	5.8	6.5	7.3	8.4	10.0
300	0.082	2.3	2.9	3.5	4.0	4.7	5.5	6.6	8.1	10.7
300	0.0446	3.0	4.2	5.3	6.1	6.9	7.6	8.4	9.3	10.4
200	0.1875	2.7	3.7	4.7	5.6	6.4	7.3	8.4	9.6	12.0
200	0.082	2.3	2.9	3.5	4.1	4.8	5.6	6.6	7.6	9.5
200	0.0446	4.7	6.1	7.2	8.3	9.4	10.3	11.9	15.0	17.0
100	0.1875	3.3	4.7	6.0	7.0	8.1	9.2	10.3	11.7	14.5
100	0.082	2.3	3.0	3.6	4.2	4.9	5.6	6.6	8.0	10.9
100	0.0446	5.4	6.8	8.3	9.6	11.0	12.3	14.0	16.0	19.0
50	0.1875	3.8	5.6	7.2	8.6	9.8	11.3	13.0	15.5	20.0
50	0.082	2.5	3.2	3.8	4.4	5.1	6.0	7.4	9.8	60.0
50	0.0446	5.4	7.4	9.0	10.5	12.0	13.5	15.0	16.9	20.0
25	0.1875	4.2	6.1	7.8	9.3	10.8	12.5	15.0	18.3	65.0
25	0.082	3.1	4.2	5.2	6.2	7.4	8.8	10.8	20.0	110
25	0.0446	5.8	7.6	9.1	10.4	11.8	13.0	14.6	16.7	20.0

TABLE IX

DEAGGLOMERATION OF CADMIUM OXIDE BY SMALL TUBES

Deagglomeration Pressure (psia)	Cumulative Size-Frequency Distribution								
	10%	20%	30%	40%	50%	60%	70%	80%	90%
	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)
0.5-in. x 0.1875-in. I.D. Tube									
414.7	1.2	1.6	2.0	2.4	2.9	3.4	4.2	5.3	7.6
314.7	1.3	1.8	2.3	2.7	3.3	4.0	5.0	6.6	11.0
214.7	1.3	1.6	1.9	2.3	2.8	3.3	4.2	5.5	8.4
114.7	1.2	1.6	2.0	2.4	2.9	3.5	4.4	5.7	8.6
64.7	1.6	2.0	2.5	3.1	3.7	4.5	5.5	7.0	15.0
39.7	2.2	3.2	4.4	5.5	6.8	9.0	16.8	38.0	72.5
0.5-in. x 0.082-in. I.D. Tube									
414.7	1.0	1.4	1.9	2.4	3.0	3.9	5.1	7.2	26.5
314.7	1.2	1.6	2.1	2.7	3.4	4.3	5.6	8.1	25.0
214.7	1.3	1.7	2.2	2.8	3.5	4.4	5.8	9.1	28.5
114.7	1.4	2.0	2.5	3.2	4.0	5.1	6.7	10.5	28.0
64.7	1.8	2.4	3.0	3.7	4.6	5.7	7.1	12.0	46.5
39.7	1.9	2.6	3.4	4.3	5.3	6.5	8.3	14.5	40.0
0.5-in. x 0.0446-in. I.D. Tube									
414.7	1.2	1.6	2.0	2.5	3.1	4.1	5.8	11.0	26.5
314.7	1.2	1.7	2.1	2.6	3.2	3.9	5.1	8.6	38.0
214.7	1.4	1.9	2.5	3.2	4.3	6.1	13.0	34.0	67.0
114.7	1.6	2.2	2.9	3.6	4.4	5.7	8.0	17.0	56.5
64.7	1.9	2.8	3.7	4.8	6.6	15.0	35.0	62.0	—
39.7	2.1	3.3	4.5	6.1	8.4	23.0	47.5	91.0	—
1-in. x 0.1875-in. I.D. Tube									
414.7	1.3	1.7	2.1	2.5	2.9	3.5	4.2	5.2	6.9
314.7	1.2	1.7	2.1	2.5	3.0	3.4	4.1	4.9	7.0
214.7	1.3	1.8	2.2	2.7	3.2	3.9	4.7	5.7	7.3
114.7	1.4	1.9	2.4	2.9	3.5	4.2	5.1	6.2	8.8
64.7	1.6	2.2	2.8	3.5	4.3	5.4	6.9	17.0	45.0
1-in. x 0.0820-in. I.D. Tube									
414.7	1.0	1.4	1.8	2.3	2.9	3.4	4.3	5.8	9.0
314.7	1.0	1.4	1.8	2.2	2.6	3.1	3.7	4.7	7.2
214.7	1.2	1.6	2.0	2.4	3.0	3.7	4.8	6.6	13.0
114.7	1.1	1.7	2.2	2.7	3.2	3.8	4.6	6.3	18.0
64.7	1.4	2.1	2.7	3.4	4.2	5.3	6.8	10.0	35.0
39.7	2.0	2.9	4.0	5.2	7.0	11.0	18.0	49.0	80.0

(Continued)

TABLE IX (Continued)

DEAGGLOMERATION OF CADMIUM OXIDE BY SMALL TUBES

Deagglomeration Pressure (psia)	Cumulative Size-Frequency Distribution								
	10%	20%	30%	40%	50%	60%	70%	80%	90%
	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)
1-in. x 0.0446-in. I.D. Tube									
414.7	1.0	1.4	1.8	2.2	2.8	3.7	5.0	7.6	22.0
314.7	1.1	1.5	1.9	2.3	2.8	3.6	4.8	7.8	25.0
214.7	1.1	1.6	2.1	2.5	3.1	3.9	5.2	7.8	27.0
114.7	1.5	2.1	2.8	3.5	4.7	6.6	15.0	36.0	—
64.7	1.9	2.7	3.7	5.3	8.4	29.0	46.0	66.0	—
39.7	2.8	5.6	12.0	30.0	48.0	—	—	—	—
2-in. x 0.1875-in. I.D. Tube									
414.7	1.3	1.7	2.1	2.5	3.0	3.5	4.1	5.1	7.0
314.7	1.2	1.5	2.0	2.4	2.9	3.5	4.1	5.2	8.0
214.7	1.2	1.7	2.1	2.5	3.0	3.5	4.2	5.3	7.4
114.7	1.2	1.7	2.1	2.6	3.1	3.7	4.4	5.5	7.3
64.7	1.4	2.1	2.6	3.2	3.8	4.6	5.9	8.2	34.0
39.7	2.1	3.0	3.9	4.9	6.1	7.5	16.0	42.0	68.0
2-in. x 0.0820-in. I.D. Tube									
414.7	1.1	1.5	2.0	2.5	3.0	3.7	4.7	6.1	11.0
314.7	1.2	1.7	2.2	2.7	3.4	4.2	5.2	6.8	11.0
214.7	1.4	1.9	2.4	3.0	3.6	4.4	5.3	6.8	11.0
114.7	1.2	1.8	2.5	3.3	4.2	5.5	7.4	14.0	39.0
64.7	1.7	2.5	3.3	4.5	6.4	14.0	38.0	60.0	—
39.7	2.6	4.5	7.8	31.0	48.0	66.0	90.0	—	—
2-in. x 0.0446-in. I.D. Tube									
414.7	1.1	1.5	2.0	2.4	2.9	3.5	4.5	6.0	11.0
314.7	1.1	1.3	1.7	2.1	2.6	3.2	4.0	5.2	7.8
214.7	1.3	1.9	2.6	3.5	4.5	6.0	8.6	21.0	47.0
114.7	1.2	1.9	2.6	3.4	4.6	7.4	21.0	35.0	57.0
64.7	1.9	2.6	3.6	5.2	8.6	23.0	32.0	42.0	54.0
39.7	2.7	5.0	23.0	38.0	59.0	64.0	—	—	—

TABLE X

DEAGGLOMERATION OF SULPHUR BY SMALL TUBES

Deagglomeration Pressure (psia)	Cumulative Size-Frequency Distribution								
	10%	20%	30%	40%	50%	60%	70%	80%	90%
	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)
2-in. x 0.0446-in. I.D. Tube									
414.7	34.0	44.0	49.0	53.0	57.0	64.0	72.0	90.0	---
314.7	7.0	25.0	37.0	44.0	49.0	54.0	61.0	72.0	---
214.7	7.0	16.0	31.0	40.0	46.0	52.0	58.0	69.0	90.0
114.7	6.0	8.0	11.0	19.0	29.0	38.0	47.0	54.0	65.0
64.7	6.6	10.0	14.0	19.0	26.0	34.0	40.0	48.0	60.0
1-in. x 0.1875-in. I.D. Tube									
414.7	5.0	7.6	10.0	13.0	19.0	30.0	46.0	57.0	66.0
314.7	4.3	6.7	9.0	13.0	23.0	34.0	42.0	54.0	70.0
214.7	6.3	9.0	13.0	19.0	28.0	38.0	48.0	58.0	72.0
114.7	6.8	10.0	14.0	19.0	26.0	34.0	41.0	49.0	62.0
64.7	7.8	13.0	18.0	24.0	30.0	38.0	45.0	53.0	64.0
0.5-in. x 0.1875-in. I.D. Tube									
414.7	4.2	5.7	7.8	12.0	19.0	34.0	45.0	57.0	---
314.7	5.3	7.8	11.0	18.0	28.0	39.0	48.0	59.0	---
214.7	5.4	8.2	13.0	19.0	22.0	35.0	43.0	51.0	64.0
114.7	6.2	8.8	12.0	16.0	22.0	31.0	39.0	46.0	58.0
64.7	7.6	13.0	18.0	24.0	30.0	36.0	43.0	51.0	61.0
0.5-in. x 0.0820-in. I.D. Tube									
414.7	8.0	18.0	31.0	42.0	50.0	55.0	60.0	68.0	80.0
314.7	9.0	20.0	31.0	41.0	48.0	52.0	58.0	65.0	76.0
214.7	8.4	17.0	28.0	38.0	47.0	52.0	57.0	64.0	73.0
114.7	9.6	17.0	25.0	35.0	44.0	49.0	54.0	61.0	70.0
64.7	10.0	17.0	25.0	34.0	43.0	49.0	54.0	60.0	72.0

TABLE XI

DISPERSION OF UNGROUND Bacillus subtilis BY MICROMEROGRAH SYSTEM

Pressure	Cumulative Size-Frequency Distribution								
	10%	20%	30%	40%	50%	60%	70%	80%	90%
(psig)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)
400 ⁺	4.5	6.3	8.2	11.1	15.7	28.5	58.0	83.0	118.0
300 ⁺	4.8	6.6	8.8	11.9	17.2	31.0	57.0	85.0	112.0
100 ⁺	4.5	6.7	9.2	13.0	22.0	44.0	66.0	88.0	118.0
50 ⁺	4.1	6.0	9.2	14.0	22.0	39.0	61.0	83.0	108.0

+ Average of 5 runs.

TABLE XII

DISPERSION OF 8.7 μ Serratia marcescens BY MICROMEROGRAH SYSTEM

Pressure	Cumulative Size-Frequency Distribution								
	10%	20%	30%	40%	50%	60%	70%	80%	90%
(psig)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)
400 ⁺	4.7	6.0	7.3	8.4	9.5	10.9	12.3	14.4	17.1
300 ⁺⁺	4.9	6.3	7.5	8.7	9.9	10.9	12.5	15.0	19.0
200 ⁺⁺	5.5	6.8	8.1	9.4	10.9	12.3	14.1	16.0	19.5
150 ⁺	5.2	6.8	8.1	9.4	10.5	11.8	13.5	14.5	18.5
100 ⁺	5.7	7.5	8.9	10.2	11.4	12.8	14.5	16.8	20.0
60 ⁺⁺	5.9	7.7	9.3	10.8	12.3	13.8	15.5	17.6	20.5
25	6.6	8.4	9.8	11.2	12.5	14.2	16.0	18.2	21.1

+ Average of 3 runs.
⁺⁺ Average of 2 runs.

TABLE XIII

DEAGGLOMERATION OF 8.7 μ *Serratia marcescens* BY SMALL TUBES

Deagglomeration Pressure (psig)	Cumulative Size-Frequency Distribution								
	10%	20%	30%	40%	50%	60%	70%	80%	90%
	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)
3-in. x 0.020-in. I.D. Tube									
400 ⁺	4.4	5.7	6.8	7.8	8.9	10.1	11.3	13.5	18.0
300 ⁺	4.4	5.8	6.9	8.0	9.0	10.1	11.5	13.8	18.0
200 ⁺	4.9	6.4	7.6	8.7	9.8	11.2	12.8	15.0	19.5
100 ⁺	5.4	7.0	8.4	9.6	10.6	12.0	13.4	15.5	18.8
60 ⁺	5.6	7.3	8.7	10.0	11.1	12.6	14.2	15.6	19.8
3-in. x 0.042-in. I.D. Tube									
400 ⁺	4.9	6.3	7.6	8.8	10.0	11.3	13.0	15.2	19.0
300 ⁺	4.7	6.1	7.5	8.8	10.2	11.3	13.0	15.2	19.5
200 ⁺	4.9	6.6	7.9	9.1	10.4	11.8	13.7	15.8	20.0
100 ⁺	5.4	7.0	8.4	9.7	11.0	12.2	13.7	16.0	20.0
60 ⁺	5.8	7.5	9.0	10.4	11.8	13.1	14.5	16.8	20.5
3-in. x 0.070-in. I.D. Tube									
400 ⁺	4.7	6.4	7.8	9.2	10.5	11.8	13.7	16.3	20.8
300 ⁺	5.3	6.9	8.2	9.6	10.8	12.3	14.0	16.5	21.3
200 ⁺	5.3	7.1	8.5	9.6	10.9	12.5	14.3	16.5	19.5
100 ⁺	5.7	7.3	8.6	9.8	11.1	12.3	14.0	16.3	20.0
60 ⁺	5.8	7.6	9.0	10.2	11.6	13.0	14.5	17.0	20.5
2-in. x 0.020-in. I.D. Tube									
400 ⁺	4.7	6.0	7.1	8.1	9.0	10.3	12.1	14.8	20.0
300 ⁺	4.5	5.9	7.1	8.2	9.2	10.3	11.8	14.1	19.0
200 ⁺	5.1	6.4	7.6	8.6	9.9	10.6	12.2	14.5	19.3
100 ⁺	5.3	6.9	8.2	9.3	10.5	11.4	13.0	15.3	20.0
60 ⁺	5.4	7.4	8.5	9.7	11.3	12.7	14.4	17.0	20.5
2-in. x 0.040-in. I.D. Tube									
400 ⁺	5.7	7.2	8.6	9.8	11.0	12.0	13.5	16.0	21.0
300 ⁺	6.2	7.9	9.4	10.5	11.6	13.3	15.2	18.4	25.0
200 ⁺	6.6	8.4	9.9	11.3	12.4	14.0	16.0	19.0	25.0
100 ⁺	6.9	8.8	10.3	11.7	12.8	14.6	16.7	19.0	27.0
60 ⁺	7.2	9.1	10.7	12.0	13.3	15.0	17.5	22.6	65.0
2-in. x 0.070-in. I.D. Tube									
400 ⁺	6.2	7.9	9.3	10.4	11.4	13.5	15.3	17.4	22.0
300 ⁺	5.9	7.5	8.8	10.0	11.1	12.2	14.0	16.2	20.0

(Continued)

TABLE XIII (Continued)

DEAGGLOMERATION OF 8.7 μ *Serratia marcescens* BY SMALL TUBES

Deagglomeration Pressure (psig)	Cumulative Size-Frequency Distribution								
	10%	20%	30%	40%	50%	60%	70%	80%	90%
	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)
2-in. x 0.070-in. I.D. Tube (Cont'd)									
200 ⁺	6.8	8.5	9.9	11.2	11.6	13.6	15.1	17.6	23.0
100 ⁺	7.0	8.8	10.2	11.6	12.7	14.5	17.0	19.3	27.0
60 ⁺	7.0	8.8	10.4	11.8	13.1	14.6	16.5	19.5	26.0
1-in. x 0.020-in. I.D. Tube									
400 ⁺⁺	5.5	7.0	8.4	9.8	11.0	12.3	14.2	17.6	34.0
300 ⁺	6.0	7.6	8.6	9.8	11.2	12.8	14.7	18.2	30.0
200 ⁺⁺	6.0	7.6	9.0	10.0	11.4	12.8	14.8	19.0	45.0
100 ⁺	6.7	8.4	9.8	11.2	12.4	14.0	17.0	23.0	90.0
60 ⁺	7.4	9.4	11.0	12.6	14.0	16.5	20.0	25.0	---
1-in. x 0.042-in. I.D. Tube									
400 ⁺	5.9	7.4	8.8	9.8	11.1	12.4	14.0	16.1	19.2
300 ⁺	6.5	8.0	9.4	10.5	12.0	13.5	15.5	19.0	60.0
200 ⁺	6.5	8.1	9.4	10.7	12.3	14.1	16.2	18.5	24.0
100 ⁺	7.0	8.7	10.0	11.5	13.3	15.2	17.5	20.0	40.0
60 ⁺	7.1	9.0	10.5	11.9	13.5	15.5	18.1	22.5	50.0
1-in. x 0.070-in. I.D. Tube									
400 ⁺	6.3	7.7	9.0	10.1	11.5	12.5	14.3	16.4	19.5
300 ⁺	6.4	8.0	9.2	10.3	11.6	13.0	14.5	17.0	24.0
200 ⁺	6.4	8.1	9.4	10.5	11.7	13.0	15.0	17.5	22.0
100 ⁺	7.0	8.7	10.1	10.4	12.5	14.0	16.1	19.5	26.0
60 ⁺	7.8	9.7	10.4	13.0	14.5	16.0	18.4	22.2	38.0

⁺ Average of 2 runs.

⁺⁺ Average of 3 runs.

TABLE XIV

BULK TENSILE STRENGTH OF VARIOUS POWDERS
FOR SEVERAL COMPACTION LOADS

Compaction Load (lb/in. ²)	Tensile Strength (lb/in. ² x 10 ³)	Relative Humidity (%)
Zinc Cadmium Sulfide		
2.24	19.4 ± 25.6%	19
1.68	12.7 ± 1.5%	19
1.13	10.4 ± 40.2%	19
0.58	7.0 ± 42.3%	19
0.25	6.6 ± 50.5%	19
<u>Bacillus subtilis</u> (Unground)		
2.24	14.2 ± 39.6%	< 1 ⁺
1.68	12.0 ± 40.5%	< 1
1.13	10.5 ± 41.4%	< 1
0.58	7.8 ± 47.6%	< 1
<u>Serratia marcescens</u> (8.7μ mmd)		
2.24	12.2 ± 35.7%	< 1
1.68	9.3 ± 18.6%	< 1
1.13	8.7 ± 29.4%	< 1
0.58	6.9 ± 27.4%	< 1
<u>Serratia marcescens</u> (3.9μ mmd)		
2.24	13.4 ± 19.3%	< 1
1.68	12.4 ± 16.0%	< 1
1.13	7.8 ± 29.4%	< 1
0.58	7.1 ± 16.3%	< 1
<u>Serratia marcescens</u> (12.6μ mmd)		
2.24	39.5 ± 7.1%	< 1
1.68	23.6 ± 3.3%	< 1
1.13	14.1 ± 13.4%	< 1
0.58	12.9 ± 0.5%	< 1
0.25	7.3 ± 8.2%	< 1
<u>Serratia marcescens</u> (Freeze dried, unwashed, unground)		
2.24	12.3 ± 20.3%	< 1
1.68	7.6 ± 4.8%	< 1
1.13	6.5 ± 19.8%	< 1
0.58	5.5 ± 12.6%	< 1
0.25	2.8 ± 25.0%	< 1

(Continued)

TABLE XIV (Continued)

BULK TENSILE STRENGTH OF VARIOUS POWDERS
FOR SEVERAL COMPACTION LOADS

Compaction Load (lb/in. ²)	Tensile Strength (lb/in. ² x 10 ³)	Relative Humidity (%)
<u>Serratia marcescens</u> (Freeze dried, washed, unground)		
2.24	10.8 ± 23.1%	< 1 ⁺
1.68	10.2 ± 9.2%	< 1
1.13	7.2 ± 16.9%	< 1
0.58	4.5 ± 9.3%	< 1
0.25	6.2 ± 13.4%	< 1
<u>Bacillus subtilis</u> (Acetone dried, unwashed, unground)		
2.24	7.9 ± 29.9%	< 1
1.68	5.9 ± 19.1%	< 1
1.13	5.2 ± 26.2%	< 1
0.58	3.4 ± 23.8%	< 1
<u>Bacillus subtilis</u> (Stokes dried, unwashed, unground)		
2.24	12.9 ± 47.0%	< 1
1.68	10.3 ± 27.6%	< 1
1.13	8.2 ± 23.7%	< 1
0.58	7.4 ± 18.6%	< 1
0.25	5.7 ± 32.4%	< 1
<u>Bacillus subtilis</u> (Acetone dried, washed, unground)		
2.24	2.4 ± 37.9%	< 1
1.68	3.1 ± 14.9%	< 1
1.13	2.6 ± 13.0%	< 1
<u>Bacillus subtilis</u> (Stokes dried, washed, unground)		
2.24	7.4 ± 50.0%	< 1
1.68	5.3 ± 35.1%	< 1
1.13	3.4 ± 21.8%	< 1
<u>Bacillus subtilis</u> (Azeotropic dried, unwashed, unground)		
2.24	6.94 ± 25.2%	< 1
1.68	4.77 ± 14.1%	< 1
1.13	4.50 ± 24.4%	< 1

(Continued)

TABLE XIV (Concluded)

BULK TENSILE STRENGTH OF VARIOUS POWDERS
FOR SEVERAL COMPACTION LOADS

Compaction Load (lb/in. ²)	Tensile Strength (lb/in. ² x 10 ³)	Relative Humidity (%)
<u>Serratia marcescens</u> (3.3 μ mmd)		
2.24	7.5 \pm 29.6%	< 1 ⁺
1.68	4.6 \pm 39.0%	< 1
1.13	3.9 \pm 15.9%	< 1
0.58	3.5 \pm 18.5%	< 1
Acrylic Resin (FT-3)		
2.24	17.1 \pm 35.4%	70
1.68	16.2 \pm 20.2%	70
1.13	13.7 \pm 26.7%	70
0.58	10.9 \pm 11.8%	70
0.25	8.9 \pm 11.7%	70
Cadmium Oxide (2.9 μ mmd)		
2.24	18.2 \pm 26.1%	70
1.68	11.4 \pm 14.5%	70
1.13	7.6 \pm 24.0%	70
0.58	6.1 \pm 14.6%	70
0.25	3.8 \pm 13.9%	70

⁺ These determinations were made in an atmosphere which was in equilibrium with a fresh surface of P₂O₅ at approximately 25°C.

TABLE XV

EXPERIMENTAL AND CALCULATED DATA FROM CENTRIFUGAL
SHEAR STUDIES OF CHARCOAL POWDER

Weight Removed per Run (g)	rpm	Radius		Loading (g/in. ²)	Shear (dynes/mm ²)
		Initial (in.)	Final (in.)		
0.0857	1810	1.00	0.63	0.0458	4.1
0.0326	3080	0.63	0.25	0.0318	3.3
0.0663	910	1.08	0.75	0.0345	0.9
0.0432	1170	0.75	0.50	0.0440	1.3
0.0478	1140	1.00	0.75	0.0348	1.5
0.0736	1600	0.75	0.50	0.0749	4.2
0.0838	1210	1.00	0.63	0.0438	1.7
0.0470	2570	0.63	0.25	0.0456	3.3
0.0437	1190	1.00	0.75	0.0318	1.5
0.0400	1710	0.75	0.38	0.0302	1.4
0.0833	1200	1.00	0.75	0.0606	2.9
0.0617	2220	0.75	0.25	0.0393	2.1
0.0580	1090	1.00	0.75	0.0422	1.6
0.0773	1720	0.75	0.38	0.0583	2.8
0.0751	960	1.00	0.63	0.0396	1.0
0.0526	1850	0.63	0.25	0.0495	1.8
0.0380	980	1.00	0.75	0.0277	0.9
0.0417	1300	0.75	0.38	0.0314	0.9
0.0383	1150	1.00	0.75	0.0279	1.2
0.0678	2290	0.75	0.25	0.0431	2.5

TABLE XVI

EXPERIMENTAL AND CALCULATED DATA FROM CENTRIFUGAL
SHEAR STUDIES OF CADMIUM OXIDE POWDER

Weight Removed per Run (g)	rpm	Radius		Loading (g/in. ²)	Shear (dynes/mm ²)
		Initial (in.)	Final (in.)		
0.1593	1950	1.18	0.75	0.0830	10.4
0.0807	2900	0.75	0.50	0.0823	15.0
0.0772	1890	1.00	0.75	0.0564	6.5
0.0581	2510	0.75	0.50	0.0594	8.1
0.1025	1900	0.88	0.50	0.0621	4.9
0.0281	2820	0.50	0.25	0.0476	4.1
0.0644	2050	0.75	0.50	0.0654	6.0
0.0817	1740	1.00	0.75	0.0594	5.8
0.0431	2350	0.75	0.50	0.0438	5.3
0.1566	3700	0.75	0.50	0.1590	4.7
0.1611	1710	1.00	0.63	0.0844	6.7
0.0577	2440	0.63	0.38	0.0735	7.1
0.1158	1930	1.00	0.75	0.0843	10.2
0.1018	1980	0.75	0.63	0.0767	8.1
0.0609	2980	0.75	0.50	0.0621	12.1
0.0380	3550	0.50	0.25	0.0646	8.9

TABLE XVII

EXPERIMENTAL AND CALCULATED DATA FROM CENTRIFUGAL
SHEAR STUDIES OF 220-MESH SILICON CARBIDE

Weight Removed per Run (g)	rpm	Radius		Loading (g/in. ²)	Shear (dynes/mm ²)
		Initial (in.)	Final (in.)		
0.7463	1250	0.75	0.38	0.5660	3.6
0.3569	1140	1.00	0.50	0.1510	4.3
0.0895	1270	0.50	0.25	0.1520	2.7
0.0656	910	0.75	0.50	0.1510	0.9
0.0479	1390	0.50	0.25	0.0815	1.7
0.1802	920	0.75	0.50	0.1400	2.6
0.0425	1340	0.50	0.25	0.0724	1.4
0.1091	960	0.75	0.50	0.0848	1.7
0.0609	1270	0.50	0.25	0.1035	1.8
0.1133	1120	0.75	0.50	0.0880	2.4
0.0947	1680	0.50	0.25	0.1610	4.9

TABLE XVIII

EXPERIMENTAL AND CALCULATED DATA FROM CENTRIFUGAL
SHEAR STUDIES OF 400-MESH SILICON CARBIDE

Weight Removed per Run	rpm	Radius		Loading	Shear
		Initial	Final		
(g)		(in.)	(in.)	(g/in. ²)	(dynes/mm ²)
0.1341	2290	1.00	0.75	0.0976	16.7
0.0722	3440	0.75	0.38	0.0545	10.5
0.0933	1790	1.00	0.75	0.0680	7.1
0.1037	3270	0.75	0.25	0.0660	7.6
0.0650	1860	1.00	0.63	0.0340	3.2
0.0412	3090	0.63	0.25	0.0399	4.1
0.0242	1600	1.00	0.75	0.0176	1.5
0.0306	2470	0.75	0.38	0.0231	2.3
0.0762	1830	1.18	0.75	0.0396	4.3
0.0466	2450	0.75	0.50	0.0454	5.9
0.0615	1460	1.00	0.75	0.0448	3.1
0.0678	2870	0.75	0.38	0.0511	6.8
0.3169	1230	1.00	0.75	0.2305	11.3
0.3064	2440	0.75	0.25	0.1950	12.3
0.3418	1350	1.00	0.63	0.1785	8.8
0.1212	2120	0.63	0.38	0.1545	11.3
0.3740	1640	1.00	0.75	0.2720	23.7
0.3505	3330	0.75	0.25	0.2230	26.9
0.1712	2000	1.00	0.75	0.1245	16.2
0.1240	2910	0.75	0.50	0.1260	23.2
0.1749	2560	0.75	0.50	0.1780	25.2

TABLE XIX

EXPERIMENTAL AND CALCULATED DATA FROM CENTRIFUGAL
SHEAR STUDIES OF 600-MESH SILICON CARBIDE

Weight Removed per Run (g)	rpm	Radius		Loading (g/in. ²)	Shear dynes/mm ²)
		Initial (in.)	Final (in.)		
0.0584	2150	0.88	0.50	0.0360	3.6
0.0088	2700	0.75	0.38	0.0066	0.8
0.0334	2280	1.00	0.50	0.0142	1.6
0.0565	2730	0.75	0.50	0.0428	0.7
0.0305	2820	1.00	0.50	0.0130	2.2
0.0177	2630	0.88	0.50	0.0072	1.1
0.0169	2510	1.00	0.50	0.0072	1.0
0.0045	2870	0.75	0.50	0.0035	0.6
0.0482	2570	1.13	0.63	0.0174	3.1
0.0281	3700	0.63	0.38	0.0060	8.0
0.0269	1570	1.25	0.88	0.0108	1.0
0.0223	2320	0.88	0.50	0.0137	1.6
0.0340	1650	1.00	0.75	0.0246	2.2
0.0212	2600	0.75	0.38	0.0160	1.8
0.0533	2400	1.00	0.50	0.0226	2.8
0.0203	2970	1.00	0.75	0.0147	4.2
0.0172	4300	0.75	0.38	0.0131	3.9
0.0174	1530	1.00	0.75	0.0126	1.0
0.0259	3600	0.75	0.38	0.0196	0.9

TABLE XX

EXPERIMENTAL AND CALCULATED DATA FROM CENTRIFUGAL
SHEAR STUDIES OF GLASS BEADS

Weight Removed per Run	rpm	Radius		Loading	Shear
		Initial	Final		
(g)		(in.)	(in.)	(g/in. ²)	(dynes/mm ²)
0.1369	3250	1.00	0.75	0.0990	34.0
0.1427	3200	1.00	0.63	0.0745	20.7
0.2810	4550	1.00	0.50	0.1190	53.4
0.0816	4900	1.00	0.63	0.0426	27.8
0.3845	3110	1.00	0.75	0.2780	87.5
0.1563	3750	0.75	0.50	0.1210	37.0
0.1921	4600	0.75	0.38	0.1460	50.2
0.0368	2800	1.13	0.75	0.0165	42.1
0.0859	4900	0.75	0.50	0.0880	45.9

TABLE XXI

SIZE DISTRIBUTIONS FOR GLASS SPHERES ADHERING TO A GLASS
SURFACE AFTER BEING SUBJECTED TO INCREASING CENTRIFUGAL FORCE FIELDS⁺

Rate of Rotation ⁺⁺	Percentage of Spheres Adhering, Having Diameters Less Than						Number of Spheres per mm ²
	10	20	30	40	50	60	
(rpm)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	
0	5.0	23.5	63.0	88.0	99.0	99.5	508
2,000	3.0	20.0	59.0	87.0	98.0	99.5	489
3,230	3.0	26.5	64.0	91.0	99.0	100.0	465
4,100	3.5	24.0	66.5	88.5	99.0	100.0	432
5,800	2.0	24.5	68.0	93.3	100.0	---	470
7,000	3.5	27.5	72.5	95.0	99.0	99.5	479
8,600	4.0	21.5	53.5	86.5	98.0	100.0	451
10,000	5.5	22.5	67.0	94.5	99.5	100.0	475
11,500	2.0	18.0	58.5	93.5	97.5	99.0	456
12,500	3.5	20.5	54.5	87.0	99.0	100.0	500
13,400	4.5	21.0	62.0	89.5	100.0	---	478
14,400	7.0	25.5	60.5	89.5	99.5	100.0	429
15,700	7.0	29.5	65.5	89.0	100.0	---	478

⁺ Laboratory relative humidity, 65 to 85 per cent.⁺⁺ Radius of rotational path, 5.7 cm.

TABLE XXII

SIZE DISTRIBUTIONS FOR GLASS SPHERES ADHERING TO A GLASS SURFACE AFTER BEING SUBJECTED TO INCREASING CENTRIFUGAL FORCE FIELDS⁺

Rate of ⁺⁺ Rotation	Percentage of Spheres Adhering, Having Diameters Less Than												Number of Spheres per mm ²
	10	20	30	40	50	60	70	80	90	100	110	120	
(rpm)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	(μ)	
0	1	5	7	16	23	28	36	41	49	59	80	97	67
600	1	5	13	23	27	33	36	45	50	70	94	100	43
1,130	2	14	21	27	33	40	46	51	64	80	94	100	40
4,000	3	18	28	37	44	52	56	67	77	96	98	100	28
7,500	7	19	36	41	50	54	62	73	85	96	100	100	10

⁺ Laboratory relative humidity, 45 to 65 per cent.

⁺⁺ Radius of rotational path, 5.7 cm.