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PHYSICO-CHEMICAL STUDIES OF SILICA OF

by

WM. H. MASSIE, B.Sc., A.R.I.C.

A thesis submitted to the University of Glasgew in fulfilment of the requirements for the Ph.D. degree in Science.

JULY, 1951.

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SUMMARY

<u>Aim of Research</u>: to develop and evaluate an apparatus for the investigation of the air-settling characteristics of silica dusts of size dangerous to health, to examine its use for the investigation of certain factors connected with dust suppression and to examine other appropriate applications of the apparatus.

1. A brief account is given of the supersedure of the 'Mechanical Theory' by the 'Solubility Theory' of silicosis, and mention is made of some practical problems associated with the measurement of dust suppression and health hazard.

2. The preparation of graded silica particles and fine mineral dusts, including sizes dangerous to health, by sedimentation from water or ethanol is described.

3. An apparatus for the examination of the air-settling characteristics of fine silica, consisting of a dust chamber into which the dust is injected and dispersed and incorporating a pair of compensated photo-electric cells connected in opposition and to a mirror galvanometer, is described. A beam of light traverses the dust cloud and is picked up by one cell; the other cell picks up the beam uninterrupted by dust. The presence of dust in the chamber therefore produces a differential current which is measured by the galvanometer.
4. Using the prepared silica dusts in the apparatus, a technique has been developed which gives the following information from <u>settling</u>

<u>curves</u> obtained by plotting galvanometer readings (converted to a percentage basis) against time:

1. Settling Factors, which for the majority of dusts, are approximately proportional to specific surface and to rates of settling of the dust clouds. Settling Factors may be used to compare different dusts or the effect of various factors on any particular dust.

2. A measure of the average terminal velocity of a dust under the turbulent conditions obtaining in the dust chamber. The terminal velocity, if the average particle size of the dust is known, gives an indication of the extent of aggregation of the particles in the dust cloud, modified by the fact that the measured terminal velocity, being affected by convection and diffusion, is lower than that which would be observed under calm conditions.

Particles of the size <u>ca</u>. 1 and 1-3 microns show considerable aggregation in the chamber but the larger grades (3-5 and 5-10 microns)show a much lesser degree of aggregation.

5. An account is given of the preparation, measurement and use in the dust apparatus, of aqueous sprays of known droplet size as dust suppressing agents and the following conclusions drawn:

1. Suppressing efficiency as measured by the Settling Factors does not change very markedly over the droplet size range 30-110 microns; the smaller droplets are slightly more effective but dust reduction is less than 50%. Dusts with particle size <u>ca</u>. 1 micron undergo the greatest reduction.

2. Under similar conditions the use of aqueous sprays containing various commercial wetting agents brings about a greater degree of reduction (60-70%).

 With water sprays only, little or no actual wetting occurs, and reduction is presumably due mainly to the 'sweeping' action of the descending spray droplets. With sprays containing wetting agents, the effect is augmented by increased wetting of the dust particles.
 Two other factors connected with dust suppression, namely, the effect of increased relative humidity, and of mixing mineral dusts with silica, were investigated.

Increased relative humidity has no effect on the rates of settling of fine silica.

The results obtained by mixing silica with certain mineral dusts of known specific surface do not confirm reports which state that the rates of sedimentation of certain mineral-silica mixtures are greater than the rates for the separate constituents of the mixtures. 7. The apparatus has also been applied to the measurement of the specific surface of small amounts (<u>ca</u>. 0.05 g.) of dust. By plotting Settling Factors against the known specific surfaces of the prepared dusts a calibration curve is prepared by means of which the specific surface of an unknown dust may be determined (accuracy, <u>ca</u>. $\pm 5\%$). The method is independent of the known decreased light extinction by particles under <u>ca</u>. 1 micron in size - a defect inherent in the usual light extinction methods of surface measurement.

Certain dusts, however, settle unexpectedly slowly, leading to overestimation of specific surface. Such anomalous dusts contained less than 50% by weight of fine particles, the balance consisting of particles with a diameter at least 5 microns greater. It is suggested that the effect is due to deaggregation of the aggregated small particles by the larger.

From the work completed it is suggested that two types of dispersion are related to every dust. One which depends on the method of dispersion and one which depends on the size and distribution of the particles and is inherent to the dust. 8. The South Wales method for the evaluation of health hazard is discussed and figures given by this method compared with Settling Factors for dusts of similar specific surface. The South Wales figures and the Settling Factors are almost identical, but the Settling Factors for anomalous dusts are considerably higher, and it is suggested that the Settling Factors give an equally effective and, in some cases, better measure of the health hazard, since they take account of dispersion in air.

9. An account is given of the methods of specific surface and particle size measurement used in the research.

10. Suggestions are made in a general discussion for future work.

INTRODUCTION

Lung disease caused by the inhalation of excessive quantities of air-borne dust, claimed 3,779 victims from the mining industry of this country in 1947. In the first six months of 1948 the number of certified sufferers was 1,584 (1). The magnitude of these figures indicates how serious is the position from the point of view of both manpower and finance.

While many types of dust when inhaled eventually produce disease, the most dangerous is silica, the inhalation of which, pure, over a period of years causes the characteristic "Classical Silicosis". The disease mainly developed by miners, and caused by inhalation of coal and rock dust, is termed "Coal-Workers' Pneumokoniosis", and is considered by many medical authorities to be a modified silicosis (2). The exact relationship between them, however, is not yet clear.

It was originally thought that silicosis was caused by the abrasive action of the sharp silica particles in the lung tissue, but this theory was displaced when it was shown by Gardner (3), that fine carborundum powder, having a hardness greater than that of silica, was inactive in the lungs of animals. It was also shown by Kettle (4), by goating silica particles with iron oxide, and by Denny, Robson and Irwin (5), using alumina as the coating, that an initially pathogenic silica dust may be rendered harmless. It may be mentioned in passing that the latter was the precurser of the aluminium treatment for the alleviation and prevention of silicosis (6).

The "Mechanical Theory" was therefore superseded by the "Solubility Theory". This theory, which is supported by a large amount of experimental data, attributes the deleterious effect of silica to a texic action which is produced in the lung tissue by solution of the silica particles in the lung fluid, causing irritation followed by fibrosis and nodular fibrosis, which, in severe cases, incapacitates the lung. This toxic action is thought also to be a deterrent to vigorous phagocytic action (the normal body mechanism for the immobilisation and/or removal of particles from the lungs) and may thus lead to excessive accumulation of dust, with eventual breakdown of the lungs or cardiac failure. The presence of silica in the lungs seems also to increase susceptibility to tubercular infection, and when infection of this type sets in the disease is rapidly fatal.

Much work has been carried out in recent years, notably by King and collaborators, on the solubility, in certain fluids, of the silica contained in mine dusts, and on the attendant effect of various factors which depress this solubility, and while it is true that, in general, the higher the percentage of silica in a siliceous dust and the greater its solubility, the greater is its toxicity, many variations from this generalisation occur, and are stressed by King (7).

The practical view is therefore taken in the mining industry that all dusts are dangerous, and should be eliminated as far as possible.

It is accepted generally that the most dangerous dusts, ignoring composition, are those of particle size less than 5 microns (8,9,10,11).

The lower limit of toxicity is less certain; but toxicity appears to increase as particle size decreases, the most pathogenic particles being those below 1 micron and down to about 20 Angstrom units when toxicity ceases (12). Van Wijk and Patterson (13), have determined the proportions of particles of various sizes removed from the air by breathing, and have found that at 5 microns removal is almost complete; at 2 microns, 80%; and at 0.2 microns, 25%. Below 0.2 microns, the percentage removed falls repidly.

The fineness of a dust is also important in determining time of suspension in the atmosphere. The finer a dust, the longer is it likely to be airborne and hence the longer is it available for inhalation. This is apart from initial concentration in the atmosphere which, if high, also increases the dust available for inhalation and must also be taken into account.

Thus, apart from research into the medico-chemical aspects of silicosis, considerable work has been carried out, particularly within the last ten years, on the many practical problems associated with dust disease.

Not the least of these has been the development of methods of dust suppression. It is self-evident that if it were possible to prevent the formation of dust or to remove completely the dust suspended in the mine atmosphere, then dust diseases would no longer exist.

The most important preventative and suppressive measures are

those of wet cutting with water or wetting solutions, foam, water infusion and spraying with various liquids and the investigations on the most effective means of application and of measurement of dust reduction have been confined almost exclusively to pratical mining conditions. From a consideration of the difficulties involved in obtaining reliable figures from measurements carried out in mine chambers, they cannot always be viewed with complete confidence. Dust concentrations fluctuate considerably within very short time limits and make it difficult and sometimes impossible to standardise conditions. Measurement of dust concentrations must be carried out using one of the "snap" sampling devices such as the Konimeter (14). the Owens Jet Dust Counter (15), the P.R.U. Hand Pump (16), or the Thermal Precipitator (17), or by gravimetric sampling (18), with the full knowledge that the "snap" samplers are not comparable with one another, and tend also to give individually erratic results, due mainly to the very small volume of the sample taken (19.20). Also. the fluctuations in dust concentration previously mentioned may materially affect even the gravimetric method, unless sampling is carried out over a considerable period of time. Particle size distribution varies from mine to mine, and even within one particular mine, and it is often impossible to say whether figures presented for reduced dust concentrations produced by suppressive metsures are, in fact, due to the reduction of all the dust in the atmosphere or merely to the reduction of any particular fraction, which, if composed of

large particles, leaves the atmosphere potentially as dangerous. This latter point is encountered particularly with the P.R.U. Hand Pump which samples a fixed small volume of air. The dust particles in the sample are deposited on a small round of filter paper, giving a spot, the light density of which gives a composite measure of the number and size of the particles in the sample. It is therefore impossible to say whether the spot is composed of a great number of fine particles or of a much smaller number of large ones. It has, however, been noted, on one occasion at least, that dust less than 5 microns in size, was suppressed in approximately the same proportion as the total dust concentration (21).

It is well substantiated that application of suppressive measures such as wet cutting during working, reduces greatly, but does not completely inhibit, the subsequent dispersion of dust to the atmosphere (22, 23, 24, 25) but the same certainty, however, does not exist of the reduction of dust already airborne, by, for example, the spraying of the suspended particles with water and wetting solutions (26).

It is improbable that it will ever be possible to reach the ideal state, and some investigators have therefore laid down figures, based on arbitrary standards, to increase the margin of safety over the incidence of pneumokoniosis. Bedford and Warner (25), for example, consider that the number of particles less than 5 microns in size should not exceed 660, and of these, not more than 600 per c.c. of

air should be coal, and not more than 60 stone. Other workers have suggested similar standards (27), and the following have been adopted by the National Coal Board (1); 850 coal (650 anthracite) particles between 1 and 5 microns, or 450 rock particles between 0.5 and 5 microns, per c.c. of air. Atmospheres containing dust in amounts greater than these standards are considered to be potentially dangerous.

A matter which has evoked considerable research in recent years, and allied to the measurement of dust concentrations, is the endeavour to obtain a measure of the "health hazard" of dusts. This is an attempt to obtain data relevant to dusts obtained from mine atmospheres, which will place them in order of the danger likely to be associated with them in the production of dust disease. It involves mainly the measurement of original concentration in the atmosphere, and an examination of the fineness of the dust. For the determination of fineness, many methods have been produced, among the more important of which are the microscopic, liquid sedimentation, permeability, and light extinction methods. Since one of the aims of the evaluation of health hazard is to facilitate comparison, it is readily appreciated that simplicity of expression is of prime importance.

The routine methods linked with the arbitrary dust concentration standards suffer from the various disadvantages pertaining to the use of snap samplers already mentioned, and in addition, are often unsuited to high dust concentrations. The gravimetric method is more suitable for obtaining a representative sample of dust which may then be examined in

the laboratory. It is increasingly accepted that the most suitable measure of the collected material is the determination of surface area, a view stressed by the acceptance of the solubility theory, since both rate of solution and solubility increase with increase in area of the particles. The most suitable method for the measurement of the surface of small amounts of material is considered by many workers to be the light extinction method, and the most recent work on health hazard using this method, is contained in the Eleventh, Thirteenth and Sixteenth Reports of the Coal Dust Research Committee (28). A method is here described for obtaining a measure of the surface area of the particles less than 5 microns contained in a dust. expressed as a weight percentage of the particles less than 5 microns contained in a "normal dust". Normal dusts are described as those which are found. by experience, to give a regularly increasing specific surface as the weight percentage of particles less than 5 microns which they contain increases.

The initial aim of the work described in the following thesis has been to develop an apparatus and technique for the laboratory investigation of the settling characteristics of silica dusts of known and sharply graded sizes, which are of importance in the production of dust disease. This entailed the initial preparation of silica dusts having the required graded sizes, The most satisfactory means for the separation of fine silica into graded fractions was found to be a modification of the liquid sedimentation method of Cummings (29).

The use of the apparatus in the investigation of certain aspects of dust suppression is described, and is followed by an account of experiments on the rapid determination of the specific surface of small amounts of dust, and a discussion of the anomalous behaviour to which this gives rise. Finally the application of this anomalous behaviour to the estimation of the health hazard is considered from its effects on the rates of sedimentation in air of small particles.

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SECTION 1

The Preparation of Graded Particles

(a) Introduction

The work described in the following sections required the preparation of considerable quantities of particles of small, known, sharply graded size, and below a maximum of 10 microns.

The methods for the separation of particles of differing sizes in a composite mixture are usually variations of the following:

1. Air or Liquid Elutriation.

2. Air or Liquid Sedimentation.

In elutriation, the fluid is mobile and carries with it particles whose sizes depend on the rate of flow of the fluid. In sedimentation, the fluid is static and the particles settle out under gravity at rates which depend on their size.

An investigation was carried out on the two methods considered most suitable, namely, air elutriation and liquid sedimentation. The intention was to separate the particles into four closely graded fractions, namely, <1, 1-3, 3-5, and 5-10 microns.

(b) Experimental

1. Starting Material

The starting material was pure Loch Aline sand (99.85% SiO_2 , density 2.65 g./c.c) ground for several days in a percelain ball mill after extraction with concentrated HCl, and finally sieved through a 200 B.S. sieve.

2. Air Elutriation

The basis of the air elutriation apparatus is a container to hold the composite dust, through which passes a current of air which removes particles below a size which depends on the velocity of the air flow. These particles then pass up a column where they are fractionated by the air stream and into a container for collection. Roller's apparatus and also that designed by Gonell, use this principle, an account of both being given by Carey (30).

An apparatus was built based on Roller's type and may be seen in Fig. 1. It consisted of a glass elutriator column 5' high and 2" diameter, to the foot of which was attached a glass cone into which the dust fell from a reservoir fitted with a vibrator. Air was blown at constant pressure through the cone and carried the dust up the elutriator tube and into a Soxhlet thimble where it was collected.

Various modifications were tried out to eliminate the main difficulties which arose in practice. Briefly these were:

1. The maintenance of a constant pressure air flow.

- 2. The elimination of fluctuations and eddy currents.
- 3. The prevention of dust adhering and collecting at various intermediate points in the tubes.

All the modifications, consisting mainly of improved methods of introducing the dust, the air, and variation of methods of agitating the dust, and finally a fractionation through cyclone tubes, failed to eliminate the defects. These were shown by microscopic examination



of the starting material to be the retention of small particles in the large sized fractions and vice versa. The fractionation proved to be extremely wide and was not clear-cut.

The method was therefore abandoned.

3. Liquid Sedimentation

The basis of this method is the calculation by means of Stokes' Law of the velocities of fall under gravity of particles of known density and known particle diameter, in a liquid of known density and viscosity. The law may be stated thus:

$$v = \frac{d^2 (r-r) \cdot g}{18 \cdot \eta \cdot 10^8}$$
 or, $t = \frac{18 \cdot \eta \cdot x \cdot 10^8}{(r-r) \cdot d^2 \cdot g}$

where t = time in seconds.

Davies (31), has calculated that at 20° C. for particles of density 2.65 g./c.c. and diameters of 50, 80 and 100 microns, settling in water, the law is applicable to within 1, 5 and 10% for these sizes respectively.

Times calculated by the author for a settling depth of 10 cms. of water at 15° C. are as follows:

Particle Diameter/

Particle Diameter.	Time.
10 microns	21.2 min.
5 "	1 hr. 24.6 min.
3 "	3 hr. 53.0 min.
l micron	35 hr. 14.0 min.

Experimental Method.

Modification of that due to Cummings (29).

The ground send available initially weighed 1,258 g. It was suspended in water containing 0.06% of Dispersol wetting agent, in a 30 litre glass tank to a depth of about 12 cm. The level of the suspension was marked and 10 cm. measured downwards and marked. The tank was then covered and surrounded by an insulating wall of air and corrugated cardboard to minimise heat fluctuations. The suspension was allowed to stand for the calculated period (35 hr. 14 min.), when the supernatant liquid above the 10 cm. mark, containing particles less than 1 micron, was drawn off into a suction flask through a glass tube which was bent at the tip so that the suspension was removed in a horizontal direction. This reduced the possibility of including in the < 1 micron fraction particles above this size. The procedure was repeated until no particles could be observed in suspension after 35 hr. 14 min., the material less than 1 micron being bottled in suspension and allowed to stand while the procedure was continued for other fractions. Seventeen repetitions were required to remove all the material less than 1 micron in size. This procedure was repeated for each of the size grades stated,

it being essential to remove all the material less than the stated size before continuing the separation in order to avoid contamination.

The next stage was the removal of the particles from suspension. For particles over 1 micron it was sufficient simply to allow the suspension to stand until the particles settled out. This also acted as a final fractionation. The clear supernatant liquor was then removed by suction, as previously described, and the resultant wet particles transferred to a vacuum desiccator to dry.

For the smallest grade (< 1 micron) settling requires a very long time (measured by weeks) and even then particles in rapid Brownian motion do not settle out. Experiments were therefore carried out on filtration through beds composed of naphthalene, a method suggested by Briscoe (32) for the sampling of airborne dust. The aim was to filter off the fine particles and to remove the naphthalene by sublimation. Several methods were tried to produce an efficient filter bed using sieved sizes of naphthalene compacted in gooch crucibles by application of a standard pressure, naphthalene crystals in alcohol, sucked through a gooch to form a bed etc., but none of these methods sufficed to filter completely the fine material and the method was abandoned.

The < 1 micron grade was finally obtained in the same manner as the others though a longer time was required; it being understood also that many very fine particles would not be included in the sample.

Details of the weights of dust obtained of each grade are given in Table 1. and photomicrographs are shown (Plates 1-4) to illustrate the comparative sizes and shapes of the particles.

The method described proved to be very efficient and gave clearcut fractions as may be seen from the photomicrographs and from the specific surfaces determined by the Lea and Nurse Air Permeability method (33) described in Section 6. This is shown in Table 2. together with figures for average particle diameters, calculated from the experimental specific surface, assuming sphericity of the particles.

From Table 2. it may be seen that the calculated particle diameter is very close to the average nominal size of each group. The agreement is considered to be remarkably good, confirming that the results obtained by the Lea and Nurse method are comparable with those obtainable by sedimentation procedures.

At this stage a quantity of a fine commercial silica dust became available and has also been used. (Suppliers: Messrs. Colin Stewart, Winsford, Cheshire). Their finest material, DRC., had a specific surface of 20,700 sq. cm./g. and a density of 2.61 g./c.c. and analysis showed 7.2% iron and aluminium impurity estimated as oxide, the remainder being silica.

4. The Separation of Mineral Dusts into Graded Fractions

For some of the experiments envisaged a supply of mineral dusts of known size was required. The minerals chosen were calcium sulphate (anhydrous), calcium carbonate and dolomite (CaMg(CO3)2 source: Duror).



<1 n Silica



Plate 2

1-3 u Silica







3-5 a Silica



5-10 a Silica

MAGNIFICATION X 1000

13 ла -

Table 1	

Figures	for the	Separat:	ion of Fi	ne Sil	ica		
17 1 84:	into Graded Fractions				t # 1978 en 1817 et		
		-					
$\sum_{i=1}^{n} e_{i} e_{i} = -i e_{i}$	<u><1µ</u>	<u>ر3-1</u>	<u>ι 3-5</u>	м	5 - 101	710,4	
Weight (g.)	48.4	49.0	111.	8	159.1	857.0	
n de la tradición de la composition de Composition de la composition de la comp	3.8	3.9	8.	9	12.6	68.1	
	Total	weight	initially	1,258	g∙		
s de la constance de la constan La constance de la constance de	11 /	12	finally	1,225	g. =	97.3%	
	Automatica Income		······································			•	
and the second sec					1	• . • •	

Table 2

Specific Surface of Graded Silica by the Lea and Nurse Air Permeability Method (Porosity - 0.5873) <14 1-34 3-54 5-104

S	Specific Surface (sq.cm./g.)	29 , 800	10,400	6 ,000	3,400
	Mean Diameter Calculated (4)	0.78	2.16	3.76	6 •46

The dolomite was ground to pass a 150 B.S. sieve; and the calcium sulphate and calcium carbonate were ordinary bench chemicals.

The aim was to separate each mineral into two fractions, namely < 5 microns and > 5 microns, the separation being checked by specific surface measurements.

Experimental Method

The method used was similar to that used above for silica except that, as less material was required, a 3 litre beaker sufficed for the sedimentation and that a non-aqueous medium had to be employed because of the slight solubility of these minerals in water. Initially kerosene was tried, but proved to be unsuccessful, since rapid flocculation of the mineral particles occurred. This is explained by Dalla Valle (34) as being due to the low adhesion-tension between the particles and the kerosene. Ethanol was then tried and found successful. Figures for these dusts are shown in Table 3.

Table 3

Specific S	Surfaces	of	Mineral	Dugte	Porceity	-	0.5873)
DDOOTTTO I		01	MTHOL GT	JUSUS !	TOTOSTUY		0.00101

Calcium Carbonate Dolomite Calcium Sulphate ` بىر5 > > 5~ > 5.1 **<**5*m* >5~ < 5 m Specific 11,000 2,700 Surface 13,700 900 17,000 2,600 (sq.cm./g.)Mean Diameter 2.1 8.2 1.7 25.2 8.6 Calculated (4) 1.3 2.80 2.85 Density (g./c.c.) 2.96

Figure obtained from subsequent experiments with the Dust Apparatus as there was not enough of this dust available for specific surface determination by the Air Permeability method.

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SECTION 2

1. The Development of Apparatus and Experimental Method

(a) Introduction

A large part of the experimental work of this thesis was the development of an apparatus for the examination of the air settling characteristics of silica particles of sizes important in the study of the silicosis problem.

Several workers have used dust chambers fitted with various devices for the measurement of the factors influencing the settling of dusts. For example, Drinker, Thomson, and Fin (35) used a 1,600 cu. ft. cabinet and followed the sedimentation of the dusts by means of a Tyndallometer; a device which measures the light reflected from a dust cloud when a beam of light is passed through it. Berkelhamer (36) used a similar method. First and Silverman (37), on the other hand, like Davies (31), measured the sedimentation by collecting samples of the settled dust at various times as sedimentation progressed on slides which were then examined under the microscope. This latter method is very slow and tedious.

Scattered.

The aim was to design a more compact apparatus for reasons of space and to reduce the amount of material required per examination. This latter point is essential when airborne mine dusts are considered, since sampling where concentrations in the mine atmosphere are low, often produces weights as low as 0.1 g.

(b) Experimental

Photoelectric Dust Sedimentation Apparatus

The design of the apparatus is based upon that used by Smellie (38), to measure the dispersibilities of mine stone dusts and further developed by Cumming, Rumford, and Wright (39), for experiments on the dust produced by various types of tetryl. These suffered from two defects: their accuracy was not great and the weights of dust required were 1 g. and 10 g. respectively, both quantities large when only a limited quantity is available.

By the inclusion of a lens, lens wiper, improved photoelectric cells, a fan for dispersing the injected dust, and a new method of analysing the results, the performance of the developed apparatus has been greatly improved. Accurate results may now be obtained with as little as 0.05 g. dust. For extremely fine dusts (<1 micron) this weight could probably be reduced even further.

2. Construction of the Apparatus

The apparatus shown in Fig. 2, is made from copper and brass, all interior polished surfaces being covered with black matt paint to reduce light reflection. It consists essentially of a dust chamber (B), of 12,167 c.c. capacity with removable base (M), into which is set the dust injection system (D), which is shown in Fig. 3. A lamp housing (F), attached to the dust chamber, contains a 40 watt lamp the light from which is transmitted through the chamber by a lens (G) which concentrates the beam and makes it more nearly parallel. The lens may





be cleaned while the apparatus is in use by means of a wiper (I), with a felt surface which may be rotated over the surface of the lens. Opposite the lens is fitted a selenium type photoelectric cell (J_1) , and opposite the lamp is fitted a similar and compensating cell (J_2) , balanced with the first. The cells are connected in opposition, through a small variable resistance (0-400 ohm.), for coarse adjustment, to a mirror galvanometer (57 ohm.), and are housed in cylinders to exclude stray light from the observation window (C), in the front of the chamber. Each cell is masked by a wall (L), containing a slit (25 x 3 m.m.), placed close to the active surface. A zero reading is obtained on the galvanometer by use of a fine adjustment (K), consisting of a thin strip of blackened foil (6 m.m. wide) which may be rotated in the path of the light beam. The circuit diagram is given in Fig. 4. Opposite the dust injector, fitted through an aperture in the roof is a three bladed fan (E), made from blackened aluminium sheet, and set in motion before the injection of a dust.

For injection of the dust a compressed air and drying train is fitted. The air may be diverted to a pressure flask (P), (approx. 600 c.c. capacity) fitted with a manometer (Q), and connected to the dust injector. There are two openings in the roof to act as outlets for the air blast, which pass to a pressure flask to trap any dust blown over by the initial blast. For humidity experiments this flask contained a dry bulb thermometer and the air continued through a further flask containing a wet-bulb thermometer.

3. Method of Operation

A weighed quantity of the dust to be examined (0.1 g. in the early experiments but later reduced to 0.05 g.) previously dried in vacuum, is placed in the injector tube, the apparatus meanwhile being brought to the required humidity (usually zero) by passage of air. A suitable zero is obtained on the galvanometer by use of the fine adjustment and the dust cylinder placed in the injector. The rubber tube from the injection system to the compressed air line is then compressed and air admitted to the flask until the pressure stands at 16 inches of mercury. A stop-clock is started and the fan switched on 5 seconds before injection of the dust. It is switched off 2 seconds after injection.

Readings of the galvanometer are taken at 10, 20, 30, 60, 90 and 120 seconds and thereafter every 60 seconds until 600 elapse, the lamp being switched on only during the taking of readings. This precaution minimises fatigue of the photo-cells and reduces to a minimum convection currents caused by heat from the lamp. The lens is cleaned between readings after the first 90 seconds.

From the galvanometer readings together with the appropriate times, it is possible to produce a settling curve characteristic of the dust injected.

The chamber is thoroughly cleaned before each experiment.

4. Presentation of Results

The basis of the method is the obscuration of light by the cloud of suspended particles measured by the indicating photo-cell. It has

been shown that the light cut off by a homogeneous suspension of particles in a liquid when a parallel beam of light is passed through it is related to the specific surface of the particles, the mean projected area of a particle of any shape when in turbulent suspension or random motion being equal to one quarter of the geometric surface of the particle (40). This will be enlarged upon in a later section. It seems reasonable that this should also apply to particles suspended in air as in a dust chamber. Since mean projected area is proportional to surface area and hence to specific surface and concentration, the galvanometer reading which is a function of the light obscured, should also be a function of specific surface and concentration. Therefore, concentration being kept constant, the galvanometer reading should then be a function of specific surface. and it should be possible to obtain an expression representative of the dust injected, which could be used to compare different dusts or different effects on one dust.

On this reasoning the graded dusts were examined in the apparatus.

Differing and distinctive curves were obtained using 0.1 g. of each dust, but it was found that two discrepancies arose. First, the curve for a particular graded dust, while recognisable from its height and shape, was not constant and it was therefore impossible to obtain a figure representative of the dust. Secondly, though the curves for 5-10, 3-5 and 1-3 micron dusts were in sequence and appeared as might be expected, those for DRC. and <1 micron dusts, instead of following

the same pattern as the others, appeared of different shape and could not be compared with them. This is shown in Graph 1. It may be seen that the initial readings for dusts smaller than 1-3 microns are very much lower than would have been expected. This is due to the fact that the light extinguished by small particles, while increasing with dimishing size of particle, eventually becomes less than the projected area of the particles would lead one to expect when the particles reach a size approaching that of the wavelength of the light passing through the suspension (41, 42). This point is illustrated effectively in Graph 2. which shows the curves obtained by plotting galvanometer readings at 60, 120 and 300 seconds against the specific surfaces of each dust (0.1 g. dispersed). It may be noted how, at 60 seconds, galvanometer reading increases with specific surface until 10,400 sq. cm./g. is reached. At this point, galvanometer reading begins to decrease as specific surface approaches that of DRC. and <1 micron silica (20,700 and 29,700 sq. cm./g.) respectively. The effect is similar at 120 seconds, but at 300 seconds much of the 1-3, 3-5 and 5-10 micron silica has settled out and the curve continues to increase after 10,400 sq. cm./g. as the relative concentration of fine to large particles increases sufficiently to mask the decrease in light extinction. Further mention of the phenomenon is made in a later section.

5. The Determination of Settling Factors

Continued investigation has developed a method which eliminates both of the above objections simultaneously since it depends on particle




size or specific surface only in so far as this is linked with rate of sedimentation.

The method is to calculate the percentage reduction in galvanometer reading as sedimentation proceeds. taking the reading at 10 seconds as 100%. From this the percentage galvanometer figure, representing the proportion of dust remaining suspended in the chamber at a certain time is obtained. By plotting these figures against the appropriate times as before, a settling curve is obtained for each of the graded dusts representative of the settling characteristics of the dusts. These curves are drawn for 5-10, 3-5, 1-3, <1 micron silica and DRC. in Graph 3. It may be observed that the curves are in their expected order. By taking from the smooth curves percentage galvanometer readings at 100, 300 and 600 seconds and averaging them it is possible to obtain an empirical 'Settling Factor' apparently approximately proportional to the settling characteristics of each dust as measured indirectly by its specific surface. Settling Factors and appropriate specific surfaces are shown in Table 4.

The Settling Factor thus gives an indication of the fineness of a dust and a comparison of the abilities of dusts to remain suspended in air. It should therefore be possible to use it to compare methods of reducing dust in an atmosphere.

6. The Measurement of (1) the Effect of Initial Dust Concentration on the Settling Curves and (2) the Error of the Apparatus

It was found possible to determine both the above simultaneously.



			Table 4			
	Spe	eific Surf	aces and s	Settling Fac	tors	y mateur (
. •		for DRC, and Graded Silica				and the lite
		۰.			en de la composición de la composición Composición de la composición de la comp	
		5-10,au	3-5,4	1-3,a	DRC .	< 1 <i>.</i>
Settlir Factor	ng	14	21	33	4 9	56
Specifi Surface (sg.cm.	.c ./g.)	3 , 400	6 , 000	10,400	20 ,70 0	29,800
			ú -		na Transista (O LE COL
				•	11月1日 金石	$\log p = \log p$
1.						
			<u>Table 5</u>			es X i j
	<u>Settli</u>	ng Factors	for Vario	ous Weights	of DRC.	
Weight DRC. (g	of ;•)	0.025	0.05	0.10	0 • 20	Maximum Difference
% Galva	n-					
(sec.)	100	77	74	76	74	3)
4. • • •	300	49	47	40	47	9 Mean
ند . بردی	600	33	28	32	29	55
Settlin Factor		53	50	49	50	4
	5 S 48 P 7 4 S 7 2	the second second				

1. The normal galvanometer reading atime curves for four different weights of DRC., namely 0.025, 0.05, 0.1 and 0.2,g. show considerable variation as may be seen from Graph 4. A factor in the cause of this variation is thought to be the speed of the fan which altered considerably during current load shedding. These curves converted to a percentage basis as previously described, are shown in Graph 5. It is apparent from this latter graph that, using the above weights of dust, initial concentration has little effect on the percentage rate of settling. This is as might be expected.

2. From these curves it is also possible to deduce a figure for the error to be expected from the apparatus. Figures taken from the curves are presented in Table 5. It is concluded from these figures that a fair error for the Settling Factor could be put at ± 2 or 3; which error may safely be considered applicable to all the dusts except 5-10 micron silica since variation tended to increase as particle size decreased from 3-5 microns to <1 micron. The 5-10 micron dust, however, gave curves which were sometimes erratic.

2. Further Development

(a) <u>Theoretical</u>

Theoretically it should be possible to calculate a value for the average rate of sedimentation of a closely sized dust as terminal velocity (abbreviated later to T.V.), by application of Stokes' Law if its average particle size is known. For sedimentation in gases





Stokes' Law is expressed as follows:

$$FV = \frac{d^2 (v - r).q}{18 \eta. 10^8}$$

where F is a constant which depends on 'd' and allows for the effect of 'slip' in gases and the other symbols have the significance stated in Section 1. The 'slip' effect occurs when the particles approach in size the value for the mean free path of the gas molecules. Values for F as given by Davies (31) are shown in Table 6.

However, while the equation applies to the sedimentation of particles in calm air (within certain limiting diameters), calm air conditions very seldom obtain and most certainly not in the dust chember or in a mine atmosphere. There, the particles are subject to more or less random movements caused by convection currents and by diffusion. These factors suffice to keep the particles evenly distributed throughout the cloud, while their overall concentration gradually diminishes by the removal of particles which settle out whnever they reach a stagnant layer which occurs near the floor and other solid surfaces. It is this diminution in concentration which is measured by the Dust Apparatus, and the smaller the particle, in general, the slower will be the reduction in concentration.

Davies has studied the problem mathematically and has produced theoretical means which, when coupled with experimental data, will give a measure of:

1. the divergence of the sedimentation of the particles from calm conditions and,

Table 6

San Andre

The slip correction, F, to multiply the velocity of spheres calculated by Stokes' Law when falling through air at 20°C. and 760 m.m. pressure.

F. akatyra a sitt Diameter (*u*) n de alter in de a 11.554. 0.02 . Exporter for the 0.04 6.106 is the state and is a on at no 2.894 in the finite of 0.10 10 the mmerical ordit volume, then the local of the person of area of the local of 0.40 1.422 est to really, the 1.00 and referen 1.164 lalos in the faile of $= 100 \text{ size in altrain 0 to } (0^{\circ} \text{ s}^{2})^{\circ} \text{ [size in a size of a 1.082]}$ 1.042 ares a be the less of the case of tien the diminuteer at aparts particulated as G. where A it. 1 area of a horizontal Section. 1.008

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 $\frac{dC/Ct}{dt} = -\frac{\partial V/h}{h} \text{ and } C = C_{c} e_{ab} + Vt/h$

2. the average terminal velocity of a dust dispersed in an air chember as opposed to that calculable by Stokes' Law from a knowledge of the average particle diameter assuming calm conditions.

It is appropriate at this point to include the theoretical proof by Davies.

Considering a cloud of particles uniformly distributed and sedimenting in an air chamber.

Supposing in time δt all particles within a distance of $V \delta t$ of the floor settle, where V = T.V.

If the numerical concentration of particles in the fluid is C per unit volume, then the number falling out per unit area of the floor is CVSt.

As a result, the mean concentration of particles in the fluid is reduced from C to (C - δ C) per unit volume.

Let h be the height of the chamber.

Then the diminution of total particles is AhSC, where A is the area of a horizontal section.

Ah&C is equal to the total number of particles which have fallen out, so that;

 $-Ah\delta c = Acv\delta t$

Whence, rate of change of concentration is

dC/dt = -CV/h and $C = C_0 exp. (-Vt/h)$

Where C_0 is the initial number of particles per unit volume and C is the average number of particles at time t.

From the above equation the number of particles in the chamber at time t was:

$$CAh = C_Ahexp. (-Vt/h)$$

Hence, the number reaching the floor up to time t is:

$$C_0Ah - CAh = C_0Ah \left[1 - exp. (-Vt/h) \right]$$

So that the number per $cm.^2$ on the base up to time t from the beginning of the experiment is:

$$N_t = C_0 h \left[1 - exp. (-Vt/h) \right]$$
(1)

From this equation:

 $dNt/dt = C_0Vexp.$ (-Vt/h), so that when t = 0, $dN_t/dt = C_0V.$

When N_t is plotted against t, this is the slope of the straight line through the origin of the curve when turbulence is absent and is the tangent at the origin to the curve when turbulence is present.

When $t \rightarrow \infty$ the curve becomes horizontal and $N_t = C_0 h$: which is again the value which would have been obtained in calm conditions.

Therefore, in calm air, two intersecting straight lines would be produced, and in turbulent air, a curve which would approach the values obtained under calm conditions at t = 0 and where t becomes large compared with h/V. This is illustrated in Fig. 5.

It is expected that as particles become larger so will they approach most closely to calm air sedimentation as turbulence will have less effect than on the smaller particles. Curves drawn $N_t \propto t$ for graded particle groups should therefore show the difference



between calm and turbulent settling.

(b) Experimental

1. The Effect of Turbulence

The procedure described has been carried out for 5-10, 3-5, 1-3 micron silica and DRC. and the curves obtained are shown in Graph 6.

To obtain these curves which show the progressive departure with decreasing size from calm settling, experiments were carried out using 0.05 g. dust each time. C_0 was calculated, assuming complete dispersion, from the average particle diameter, which was itself calculated from the experimentally determined specific surface. N_t was calculated from a knowledge of C_0 , h (the height from roof of the chamber to point of measurement), percentage galvanometer reading, and the volume of the chamber.

The procedure is illustrated below for 1-3 micron silica.

Specific surface	1	10,400 sq.cm./g.			
Average particle diameter		2.16,4			
Volume of chamber	1	2,167 c.c.			
h	1	1.5 cm.			
Determination of Co:					
Volume of 2.16 particle	T	5.278 x 10 ⁻¹² c.c.			
Weight (vol. x 2.65)	N	$14.05 \times 10^{-12} g.$			
No. of particles in 0.05 g.	-	$(0.05 \times 10^{12})/14.05$.			
No. of particles per c.c. (Co)=	$(0.05 \times 10^{12})/(14.05 \times 12,167)$			

$$C_0 \equiv 293, 900$$

and

 $N_t = [(\% \text{ G.R. at time t})/100] \times C_0 \times 11.5$

where G.R. = galvanometer reading.

The N_t figures for DRC., 3-5 and 5-10 micron dusts were converted proportionally to the same initial numerical basis as the 1-3 micron grade. Thus, 0.05 g. 1-3 micron silica produces in the chamber 293,900 particles per c.c. 0.05 g. DRC. produces 2,325,000 particles per C.C., which is approximately 10 times 293,900. Therefore, in order to permit the plotting of the figures for these two dusts on the same scale for comparison, the original N_t figures for DRC. were divided by 10. The procedure for the 3-5 and 5-10 micron silica was similar except that the original N_t values were multiplied by 5 and 30 respectively.

The curves in Graph 6. show that the 5-10 micron grade approximates most closely to calm air settling, while it may be observed that DRC. shows a very great departure.

2. The Calculation of Terminal Velocity

From equation 1. it may be seen that:

$$t/\log_{e} (1 - N_t/C_0h)^{-1} = h/V$$
,

showing that a straight line should result, passing through the origin, when t is plotted against the log expression. From this, it should be possible to calculate the average terminal velocity of the dust dispersed in the chamber by measuring the slope of the line and equating with h/V, of which h is known (ll.5 cm.). In practice, the lines are sometimes broken and curve downwards to the origin owing to the settling out of the



larger particles. On these occasions the maximum and minimum slopes are taken to give the terminal velocities of the maximum and minimum particles in the dust, and the average slope to give the average terminal velocity.

Davies calculated N_t by counting and measuring the particles deposited on a slide on the base of the chamber and, while this is probably the most accurate method it is very slow.

Using the Dust Apparatus, it is unnecessary to calculate N_t , and the \log_{Θ} expression may be derived from the percentage galvanometer readings since N_t and C_0 occur in the expression as a ratio. N_t may be calculated only if C_0 is known and this entails the prior knowledge of specific surface or average particle diameter.

The technique has been applied to 5-10, 3-5, 1-3 micron silica and DRC., and the curves obtained are shown in Graph 7.

Both DRC., and 1-3 micron dusts give a single straight line while the curves for 3-5 and 5-10 micron dusts are broken, each in one place. Figures calculated for these dusts are presented in Table 7.

It may be noted from these figures that the actual terminal velocity of DRC. is greater than the value calculated from Stokes' Law, which seems to indicate that this dust is aggregated to a considerable extent during sedimentation. The aggregation is probably greater than is indicated by the average particle diameter calculated from the experimental terminal velocity owing to the effect of turbulence in decreasing the rate of sedimentation.



Table 7

Calculated and Experimental Terminal Velocities for Graded Silica

Dust	Av. Particle Diam. calc.	T.V. calc. from Stokes'	T.V. from Graph		Av. Particle Dia. appropriate to -	
	from Sp.Surf. (AL)	Law. (cm./sec.)	vı	v ₁₂	v_	V ₁₂
DRC.	1.09	0.0108	-	0.0227	-	1.58
1÷3,4	2.16	0.0402	-	0.0427	-	2.23
3 - 5,4	3.76	0.1171	0.1085	0.0572	3.62	2.63
5-10 ,u	6.46	0.3414	0.2210	0.1210	5.20	3.88

T.V. - Terminal velocity.

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V1 - T.V. of maximum sized particles.

V₁₂ - Average T.V. of group.

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The figures for 1-3 micron dust show a similar trend but to a less extent.

3-5 and 5-10 micron dusts, on the other hand, give experimental terminal velocities below the calculated values indicating that aggregation, if it occurs, is not sufficient to mask the effect of turbulence.

3. Conclusions

An apparatus has been developed which is suitable for the examination of the sedimentation of dusts of size up to between 5 and 10 microns.

From experiments with the apparatus it is possible to obtain three items of information:

1. A 'Settling Factor' which is apparently representative of the settling characteristics of the dust dispersed, and which may be used to compare different dusts, or the effect of various factors on the sedimentation of an individual dust.

2. A rough qualitative guide to the effect of turbulence on the sedimentation of a particular dust.

3. An estimation of the average terminal velocity of the particles in a dust, which, in turn, for a dust whose average particle diameter is known, indicates the extent of aggregation of the particles in the cloud.

From the work completed it also appears that two types of dispersion are encountered:

1. One which is variable and depends on the weight of dust injected and on the speed of the fan, and which varies to a greater extent for the smaller closely graded dusts than for the larger or more widely graded dusts. This is considered to be due to the removal of some of the dust injected by the blades of the fan and on the roof of the chamber. This is indicated by the varying curves obtained by plotting actual galvanometer reading against time.

2. One which depends only on the size range of the dust injected and appears to be constant for a particular dust. Thus, the curve percentage galvanometer reading \propto time for any dust is constant and indicates that the particles of the dust are broken up into similar proportions of discrete and aggregated units each time it is dispersed. This point will be enlarged upon later.

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SECTION 3

The Investigation of Certain Factors Connected with Dust Suppression.

1. The Effect of Increased Relative Humidity on the Sedimentation of Graded Silica

(a) Introduction

This investigation arose owing to information received on the incidence of pneumokoniosis in two coal pits in Stirlingshire. These pits. A and B, lie within a few miles of each other and work approximately the same geological strata. Pit A is comparatively free from cases of dust disease, only two certified cases up to 1950 having been reported. From B, however, a much greater number of the workers have been certified. While little information on comparative quantitative dust concentrations is available, it is evident to the naked eye that A is much dustier than B. In addition. A is a dry pit and humidity conditions are greatly less than saturated. whereas B is very wet and recent measurements of humidity made by the staff of the Mining Dept., Royal Technical College, Glasgow, gave a value of 100% relative humidity at the bottom of the upcast shaft. In all the working places the air is completely saturated, and in many a distinct mist may be observed. The strata are naturally damp and in many of the road-ways water is found in pools on the floor.

The problem was to determine whether fine particles might be preferentially maintained in suspension in the atmosphere because of increased humidity. It was considered, therefore, that by examining graded silica dusts at both 0% and 100% relative humidity in the Dust Apparatus, data would be obtained indicating whether or not increased relative humidity had any effect on the sedimentation of the finer silica particles.

Drinker, Thomson and Fin (35), in a paper on the stability of dust, fume and smoke clouds, examined, among other things, the effect of increased humidity on silica particles of median size 1 to 3 microns and ranging from 0.5 to 30 microns, but could draw no positive conclusions from their results.

Bedford and Warner (25), in a comprehensive survey of comparative humidity and dust conditions in certain South Wales collieries, state that collieries having a high incidence of pneumokoniosis are generally reported as being deeper and drier than those of low incidence. This statement does not agree with the reports mentioned on A and B, but should be taken as the general rule. Their experiments, however, indicate that while there is a tendency, according to particle counts, for the drier mines to be the dustier, there is no evidence to show that dust concentrations within a mine vary inversely as the atmospheric humidity. They suggest, rather, that reduced dust concentrations in wet pits are due to the removal of dust by adhesion to wet surfaces of the coal and rock.

(b) Experimental

The method was to obtain settling curves for the five graded silica dusts (0.1 g. injected) at both 0% and 100% relative humidity and to

compare them. Any increase or decrease in settling rate would immediately be obvious from the settling factors.

0% relative humidity was obtained by blowing dry air through the chamber for about 20 minutes, checking with the wet-dry bulb thermometers.

100% relative humidity was obtained in a similar manner using watersaturated air.

The normal G.R.∝ time curves obtained are shown in Graphs 8 and 9. These curves suggest that the initial dispersion at 100% relative humidity is decreased greatly for DRC. and <1 micron silica but decreased to a lesser extent for 1-3 and 3-5 micron silica, and not at all for 5-10 micron silica. It is thought that this is due mainly to increased removal on the damp fan and roof of the chamber.

The % G.R. \propto time curves are drawn in Graphs 10 and 11 and show that no significant difference occurs between those at 0% and 100% relative humidity, for any dust.

Settling Factors taken from these curves are given in Table 8.

The figures for 5-10 micron silica are slightly erratic but not, it is considered, enough to alter the conclusion. This error is thought to arise owing to the decreased sensitivity of the indicating photo-cell at the low impulses transmitted by this dust.

New photo-cells were subsequently obtained and the settling factor for 5-10 micron silica found to be 5, the others being substantially unaltered.









Table 8

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Settling Factors for Graded Silica at 0% and 100%

Relative Humidity

0% R.H. 100% R.H. • Indiad Street 49 50 - 9 azā 35.00 - 1 n na statu na haine anne 30 - the the fight the of Conthemant in the mines, especial - remarking the remarking the destance dueta - Guarlete recevel - resplictable and statical particular of allos sector from here been Layrand Bassidermbis curpless. They fall into in .

the hass for the prevention of the behaving authorne. The hade for the removal or reduction of duct already airborne and of the experimental work has been contined to group 4, The haded sater influeion, the spropuls of lossing basis and dr the water and worthing solutions etc., ast suffling, and so a

(c) Conclusions

It is concluded that increased humidity in the dust chamber is without effect on the sedimentation of silica dusts of particle size 1 micron to between 5 and 10 microns, apart from decreasing the initial dispersion of the smaller dusts.

2. The Effect of Sprays on the Sedimentation of Graded Silica

(a) Introduction

Silicosis and pneumokoniosis are diseases of the lungs caused, as previously stated, by excessive inhalation of dust, which is considered to be most dangerous at particle sizes below about 5 microns.

Owing to the high rate of disablement in the mines, especially in South Wales, considerable research work has been carried out on methods for the removal or reduction of air-borne dust. Complete removal is as yet impracticable and various methods of dust reduction have been devised, some having considerable success. They fall into two general classifications.

A. Methods for the prevention of dust becoming airborne.

B. Methods for the removal or reduction of dust already airborne.

Most of the experimental work has been confined to group A, and has included water infusion, the spraying of loading banks and drill tips with water and wetting solutions etc., wet cutting, and so on; and as much as 50 to 70% reduction of dust dispersed has been obtained (43).

Experiments in group B have included the spraying of airborne dust concentrations with water and wetting solutions and a similar degree of

reduction claimed (26).

In addition, the effect on sedimentation of mixing certain mineral dusts with silica has been reported (35, 36, 37). This is dealt with in the next part of this section.

According to the literature, while it is well established that wetting of the coal or rock before and during working reduces greatly the dust subsequently dispersed to the atmosphere (45, 25), the evidence regarding the removal of dust actually airborne by, for example, spraying and other means, is not so conclusive. Little work has been carried out under strictly controlled laboratory conditions. The use of wetting agents appears to reduce the amount of water required to give minimum dispersal of dust but there is no conclusive evidence that wetting agents might give more effective reduction of dust already airborne (26). In one case which has been reported, however, reduction of airborne concentrations in an experimental mine gallery were found to be 76% for water sprays and 88% using wetting solution sprays (46). It is unnecessary to comment upon the small difference between these two figures.

This second part of Section 3. isconcerned with group B and, in particular, the reduction of airborne silica dust by spraying.

The trend in recent years in the design of sprayers has been to enable the production of as small droplets as possible. This has more recently given way to sprayers based on the main requirement that the droplets emitted should be fairly uniform and contain the proportion

of about 50% of droplets 15 to 25 microns in size (47).

The mode of action of the spray droplets on the suspended dust particles is not known, the general opinion being that the droplets wet the dust particles and by increasing their weight and size cause them to settle more rapidly. The view has also been expressed (48) that a sweeping action caused by the passage of the spray droplets brings down particles which are thought to be too small to be efficiently wetted.

The initial preferencefor very small droplets was presumably due to an assumption that the smaller the spray droplets, the more easily would they wet the dust particles. This accepts the view that, as a liquid droplet decreases in size so will its surface tension decrease. This has been shown theoretically to be true, but only for droplets of very minute dimensions (49) and is therefore not applicable to droplets of the size normally emitted from sprayers.

The work in this part of Section 3. attempts to throw light on four points:

- 1. the effectiveness of water sprays for the reduction of airborne silica dust,
- 2. the optimum size of spray droplet related to size of silica dust particle.

3. the effect of sprays of wetting solutions,

4. the mechanism of the action.



(b) Experimental

The work required; 1, silica dust of known, small closely-ranged particle size; 2, a means of observing the sedimentation of these dusts in air; and 3, a means of producing spray droplets of measured sizes.

1 and 2 have already been described. 3 follows.

1. The Production and Measurement of Small Spray Droplets

Two basic types of sprayer were developed; A, a commercial plunger type and B, made from glass on the perfume sprayer principle. They are shown in Fig. 6.

From each type two spray jets containing droplets of different size range could be produced: A1, by normal operation, A2, by slow operation, B1, by normal operation, and B2, by normal operation with the air bleed hole closed.

The measurement of the droplets emitted by the sprayers presented a difficulty for two reasons. First, when the droplets were collected on glass slides previously coated with paraffin wax, for microscopic examination, it was found that the mmaller droplets evaporated so quickly that exact measurement was impossible. Second, the droplets on the slide were not spherical but hemispheroidal. While it is possible to introduce a mathematical factor for conversion to spherical diameters (50), it was considered that a direct measurement technique was more desirable.

The method used was suggested by Doble (50), and used later by Dimmock (51) and consists of collecting the spray droplets in the dust

chamber in a shallow petri dish containing a small quantity of a mixture of liquid paraffin and kerosene (4 : 1 by volume). In this liquid the spray droplets, which are immiscible and penetrate the surface, retain their spherical shape, do not evaporate, and may easily be measured and counted microscopically.

A total of about 200 droplets was measured in several different fields for each spray and the results are given in Table 9.

In the following experiments the sprays are injected into the chamber through an aperture near the top: of the front side of the chamber. The aperture may be plugged when not in use.

2. Water Sprays

Initial experiments were carried out using sprays A_1 and B_1 . <u>Ca</u>. 0.28 g. of each was separately injected at 30, 180, 300 and 420 seconds into dispersions of each graded dust (initial weight 0.1 g.) giving a total weight of <u>ca</u> 1 g. spray.

Settling curves for spray A₁ with curves for the unsprayed dusts for comparison, are drawn in Graph 12. Curves for sprayed 5-10 micron silica were erratic and have been omitted.

The curves show that no significant effect has been produced by the injection of the sprays.

The quantity of water injected was then increased by a factor of five and <u>ca</u> 1.4 g, of sprays A₁ and B₁ injected as before, giving a total weight of <u>ca</u> 5.6 g. Curves were again drawn, and those for DRC. are presented in Graph 13.

Table 9

Size Frequency of Spray Droplets

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		Frequency %				
Spray	Al	Bl	A2	B2		
10 - 50,4	33	79	14	90		
50 -1 00,u	4 6	15	39	8		
100-200 ₁	21	6	47	2		
Mean Droplet						
Size (4)	74	39	110	30		
			195 			
	an An An Anna Anna An	1 1 1 1 1	an a			




They indicate no dust reduction using spray A_1 but a small amount with spray B1, namely, a difference of 10 in Settling Factor (about 20%). The curves for the other dusts were similar to those in Graph 12.

It may be observed that in these experiments the sprays were injected intermittently, so that it might be concluded that the effect of each spray injection would have disappeared before the next was introduced. Consequently to produce an increased effect, sprays A1 and B1 were injected continuously for a short period. The dusts were dispersed in the usual way and an initial galvanometer reading obtained. <u>Ca</u> 0.28 g. of spray was then injected every five seconds for periods which depended on the size of the dust particles, being greater with the smaller dusts and also coinciding with the initial high readings of the galvanometer. A final galvanometer reading was obtained after the sprays had been injected. The procedure was repeated without spray injection and the figures obtained are shown in Table 10.

Because of this method of introducing the sprays it was impossible to obtain settling curves on this occasion. The figures presented are consequently not comparable with those obtained from previous and later curves, being a percentage reduction in galvanometer reading during spraying over a certain period compared with percentage reduction in galvanometer reading without spraying during the same time interval.

The figures in Table 10 show that only with DRC. and <1 micron silica is there any increased reduction of dust, as measured by the

Dust	G.R. @ secs.	Total wt A _l	of Spray	% Re No	ducti Spra	on in Y	G.R Al		en ti Bl	mes stated
- -	the second second								-	
DRC .	10 & 300	12.9	11.5	47) 52) 59)	53		74) 74) 76)	74	86) 73)	79
< 1,4	T #	12.9	- -	48) 47)	4 8		70) 70)	70	-	
1-3,4	120 & 300	2,445 6 7 - 1	5.8	66) 76)	71	11.2	75) 70)	~.73	63	
3 - 5,4	60 & 180	5.0 5.0	4.5	70) 74)	72		71) 70)	71	69) 72)	71
ير 5 -1 0	10 & 90	ener 7 nan sa 2.₀8 andas 1 na	est up it ist en est interim	64) 68)	5 66		63) 68)	66		с <u>х</u>
	a sertagur	an a	e per en	5	1. J. 1		A Ta	turen dege	4., MA	

Continuous Spray Injection

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percentage galvanometer readings, due to the effect of sprays. It amounts to about 20 - 25% between 10 and 300 seconds. It may also be noted that because of the longer time available for injection, a greater weight of water was introduced. For the other dusts, the results indicate no reduction, with the possible exception of 1-3 micron silica and spray B_1 .

Since it appeared that continuous spraying might be effective and that more than 1.4 g. spray per injection in intermittent spraying would be necessary, the following compromise was adopted.

<u>Ca</u> 2.8, 5.6, and 8.4 g. respectively of sprays A₁ and B₁ were introduced separately in injections of 2.8 g. into each dispersed dust at 30 seconds, 30 and 60 seconds, 30, 60 and 90 seconds, producing three curves for each dust. The settling curves for DRC. and 3-5 micron silica are shown in Graphs 14 and 15, 16 and 17. Settling Factors taken from these curves and from the curves for <1 and 1-3 micron silica are presented in Table 11.

It is again obvious from these figures that reduction is not large but is greatest for DRC. and < 1 micron silica. A tendency toward greater reduction with spray B_1 may also be observed.

The manner of injection of the above sprays means that spray particles in the chamber from the first injection still exist during subsequent injections. This produces a sudden increase in the galvanometer readings taken immediately after injection and accounts for the occasionally erratic nature of the figures. It is also





Table 11		

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Dust			DF	RC .					~ ;	مرا	
Spray		Al			Bl			Al			E
Injections	1	2	3	l	2	3	l	2	3	l	
Settling Factor	39	38	36	29	27	26	51	54	41	43	32
Reduction	10	11	13	20	22	23	6	2	15	13	24
% Reduction	20	22	26	4 0	44	46	10	3	26	23	4
-											
Dust			1-3	3 m					3-	5 <i>.</i>	
Spray		Al	•		Bl			Al	•		I
Injections	1	2	3	1	2	3	l	Ż	3	l	
Settling Factor	36	32	29	35	30	24	21	21	21	20	1
Reduction	-3	1	4	-2	3	9	0	0	0	i	l
% Reduction	-	-	12	-	10	27		-	-	-	2

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considered that the true dust reduction after injections 2 and 3 is, in fact, greater than that observed experimentally being partially obscured by the presence of spray particles in the initial stages of the experiments.

Conclusions

Summing up these experiments, it appears that dust reduction, as measured by percentage reduction of the Settling Factors, is not great, but is greatest for the smaller dusts and with an intense injection of water sprays A_1 and B_1 , of which B_1 has slightly the greater effect. An explanation is later suggested for these results.

3. The Effect of Spray Droplet Size

To determine whether droplets of sizes both larger and smaller than A_1 (74 microns) and B_1 (39 microns) would be more or less effective in reducing dust, sprays A_2 (110 microns) and B_2 (30 microns) were, in addition, used in the following experiments.

<u>Ca</u> 8.4 g. of A₁, A₂ and B₁ was introduced in injections of <u>ca</u> 2.8 g. at 30, 60 and 90 seconds into dispersions of DRC., < 1, 1-3 and 3-5 micron dusts. Owing to the time involved, spray B₂ could be injected once only (<u>ca</u> 2.8 g. at 30 seconds) as the quantity of water ejected per operation of the sprayer was very small.

Curves for these experiments are shown in Graphs 18, 19, 20 and 21, and figures from the curves given in Table 12.

The figures and the curves show what appears to be a gradually increased reduction of dust as the droplet size of the spray decreased,





again more noticeable with DRC. and < 1 micron silica.

It is advisable to note a this point that while the Settling Factors have an error of ± 2 or 3, when they are converted to percentage reduction the error is considerably increased and by amounts depending on the dust of the order times 2, 1.5, 3 and 5, for DRC., < 1, 1-3 and 3-5 micron dusts respectively.

4. Wetting Solution Sprays

Three wetting solutions were used as spray A₁ which was easiest to manipulate. The injection of the spray was standardised at <u>ca</u>. 8.4 g. (2.8 g. per injection) at 30, 60 and 90 seconds as in previous work.

The solutions were as follows: concentration 1% by weight in each.

1.	Non ionic:	Lissopol N.	(ethylene oxide condensate.)
2.	Cationic :	Lissolamine A.	(cetyl trimethyl ammonium bromide.)
3.	An ic inic :	Lissopol A. T.	(probably a long chain sodium sulphonate.)

Suppliers: Imperial Chemical Industries Ltd.

The results obtained are shown in Table 13. and the curves in Graphs 22, 23 and 24, with those for water sprays for comparison. < 1 micron silica has been omitted as it shows effects similar to DRC., but less in extent.

It may be observed that the use of wetting solutions has produced an increase in dust reduction over that obtained using water sprays.





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Water Sprays A1, A2, B1 and B2 on Graded Silica

Dust	DRC .			<1.u				1 - 3 <i>m</i>			3-5,4					
Spray	A2	Al	^B 1	B2	A2	Al	Bl	B2	A2	Al	Bl	B2	A2	Al	Bl	B2
Settling Factor	40	36	26	24	50	41	39	38	31	34	24	24	16	21	16	9
Reduction	9	13	23	25	6	15	17	18	2	-1	9	9	5	0	5	12
% Reduction	18	26	4 6	50	10	26	30	32	6	-	2 7	27	25	5	25	60

Table 13

Wetting Solutions Sprays on DRC., 1-3 and 3-5, Silica

Dust	DRC .				يىر3- 1				ى <i>مر</i> 5 - 3			
Wetting Solution	Nil	1	2	3	Nil	1	2	3	Nil	l	2	3
Settling Factor	36	16	17	19	29	11	11	10	20	5	7	8
Reduction	13	33	32	30	4	22	22	23	ĺ	16	14	13
% Reduction	26	66	64	60	12	6 6	66	69	5	79	69	6 4

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Reduction of DRC., increased about 2.5 times from 13 to 32; 1-3 micron silica about 5.5 times from 4 to 22; and 3-5 micron silica about 15 times from 1 to 15. There is no evidence that one wetting agent is more effective than another. It is also apparent that the percentage reduction in Settling Factor for each dust is comparable when wetting solution sprays are used, but, as was previously indicated, the reduction with water sprays decreases as the particle size of the dust increases.

(c) <u>Conclusions</u>

1. The Effectiveness of Water Sprays

The reduction of graded silica dusts by spraying with water is greatest with dusts about 1 micron in size and decreases as the particle size of the dusts increase, varying from about 5 to 50% depending on the spray and weight of water used. It is thought, however, that dust particles in rapid Brownian motion are probably unaffected by the sprays. This is suggested from observation during the experiments, the flattening of the latter portions of the fine dust curves, and from the smaller reduction obtained with <1 micron silica compared with that for DRC., (1.09 micron).

2. The Optimum Size of Spray Droplet

There is no evidence that any particular size of spray droplet is the most effective in reducing dust of any particular size, but results indicate a tendency for dust reduction to increase as the droplet size of the sprays decreases. Again, while reduction is not

large, it is most noticeable with DRC., amounting to between 18 and 50%.

3. The Effect of Wetting Solutions.

Wetting solutions appear greatly to increase the reduction of dust, and by a greater percentage for the larger dusts, over that obtained with water sprays. Thus, from 26 to 64, 12 to 66 and 4 to 49% for DRC., 1-3 and 3-5 micron dusts respectively.

The various wetting agents are equally effective.

4. The Mechanism of the Action

Using water sprays it may be considered that the mechanism is mainly the mechanical action of the sprays on entry into the chamber, reduction being caused by the impact and sweeping action of the spray droplets as they are injected and settle out. This would explain the greater effect of the sprays on 1 micron particles while it is unlikely that particles in rapid Brownian motion would be affected.

Also, it might at first be thought that the larger spray droplets would be the more effective; but it must be remembered that, in addition to the greater numbers of small droplets in a fixed weight of spray, the smaller sprays are injected with greater force owing to the type of sprayer used.

It is obvious, however, that while this might explain the effects observed with water, it cannot explain those observed with wetting solutions, which produce a greater reduction to all intents similar for each dust (64-70%).

It must be concluded, therefore, that wetting occurs. If this is true it is impossible to exclude the possibility of wetting by water sprays. Therefore, it is considered that both actions occur but that the wetting is greatly increased with the wetting solutions.

5. Practical Considerations

It must be emphasised that the above conclusions are based on experimental data obtained with a dust chamber having a capacity of 12,167 c.c. and it would be necessary to exercise caution in translating them to practical mining conditions.

Among the more obvious items which would require to be considered are, 1. the greater turbulence which undoubtedly exists in a mine chamber, 2, the differing types of dust particle which might be encountered in a mine (coal particles are notoriously difficult to wet), 3, the fact that dust particles in a mine atmosphere are probably already wet and surrounded by a film of moisture, and 4, the much lower dust-concentrations encountered, except in the vicinity of loading banks, drill tips etc., as compared with those of necessity used in the Dust Apparatus.

3. The Effect on the Sedimentation of Silica of Mixing with Certain Mineral Dusts

(a) <u>Introduction</u>

From time to time, as mentioned in the previous part of this section, various workers have investigated the use of mineral dusts as modifying agents in the sedimentation of silica with a view to

increasing the rate of sedimentation and hence reducing the amount in the mine atmosphere available for inhalation. Dod and Lewis (44), give an account of experiments carried out in Kalgoorlie and state that they have been able to remove almost all the suspended siliceous dust from a mine chamber in a comparatively short period of time, (ca. 15 mins.), after it had been produced by shot-firing. The basis of their method was to tamp the explosive charge with a mixture of limestone and magnesite. They attribute the removal of the siliceous dust to the neutralisation of the electrostatically charged particles

of silica by oppositely charged mineral and subsequent flocculation.

Berkelhamer (36), followed this work with experiments in the laboratory using a dust chamber, but failed to correlate the charges of various minerals with their effects on the sedimentation of silica. The most important effect obtained was that the addition of calcined gypsum to quartz of small particle size increased the rate of sedimentation of the mixture over that for either component. Limestone gave a similar effect, but with some other dusts the mixtures settled more slowly than either component. Berkelhamer was unable to suggest an explanation of r these results.

First and Silverman (37), then investigated the settling characteristics of silica-calcium carbonate (pure limestone) mixtures and came to the conclusion that no significant acceleration in the settling rate of the silica mixtures occurred, as measured by lightfield microscopic technique.

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In an attempt to elucidate further the conflicting evidence presented above, experiments were undertaken with the Dust Apparatus.

(b) Experimental

The three minerals used were calcium sulphate, dolomite and calcium carbonate, sedimented from alcohol as specified in Section 1, each into two fractions, namely, >5 and <5 microns, with specific surfaces as follows:

	>5 _u	< 5 <i>1</i> 1	
calcium sulphate,	2,600	17,000	
dolomite,	900	13,700	
calcium carbonate,	2,700	11,000 sq.cm./	g.

The silica dust used was DRC., specific surface 20,700 sq. cm./g.

Settling curves were obtained in the usual manner for each dust using 0.05 g. material, and also for mixtures of the dusts with DRC., in the proportion of 1:1 by weight. These curves are shown in Graphs 25, 26 and 27. Terminal velocities were calculated from the curves in Graphs 28, 29 and 30.

Results

1. > 5 micron Mineral-Silica Mixtures

The results are shown in Table 14. The figures in brackets are the averages of the figures for the components of the mixtures; that is, values which might theoretically have been expected.













The experimental results, however, show that the settling rates of the mixtures are actually less than those calculated, as indicated by the high Settling Factors and the low terminal velocities.

Considerable speculation arose on the reason for the phenomenon and it is later in this thesis explained by effects related, not to the chemical composition of the minerals, but to the altered particle size distribution on mixing with fine silica, as similar figures may be obtained by mixing DRC. with 5-10 micron silica. See Table 15. This is enlarged upon in the following section.

2. < 5 micron Mineral-Silica Mixtures

The figures for these experiments are as shown in Table 16.

It may be observed that the Settling Factor calculated for the calcium carbonate-silica mixture from the average of the Settling Factors of its components is comparable with the experimental value. Those calculated for dolomite and calcium sulphate-silica mixtures, while not identical with experimental values, are nevertheless not sufficiently different to warrant the assumption that an increase in the rate of sedimentation has occurred, although the discrepancy is only just outside the experimental error. Furthermore, an examination of the terminal velocities of the mixtures shows that the experimental values are very close to the values calculated for the components of the mixtures. The differences between experimental and calculated values prove to be 0.0015, 0.0071 and 0.0084 cm./sec., for calcium sulphate, dolomite and calcium carbonate mixtures

>5~ Mineral + DRC. Mixtures

Dust	Settling Factor	Average T.V. (cm./sec.)
DRC.	52	0.0227
Ca B 04>5~	3	0.2182
* * +DBC.	51 (28)	0.0224 (0.1205)
Dolomite > 5,4	5	0.2093
* + DRC.	. 39 (29)	0.0358 (0.1160)
CaCO3>5	4	0.1480
• • + DRC .	53 (28)	0.0160 (0.0854)
	-	,

Table 15

5-10- Silica + DRC. Vixtures

Dust	Settling Factor	Average T.V.
DBC.	52	0.0227
5-10 ₄ 4	5	0.1210
DRC. + 5-10. (1:1 by vt.)	46 (28)	0.0302 (0.0718)

< 5. Mineral + DRC. Mixtures

Dust	Settling Factor	Average T.V.
DRC.	52	0.0227
CaSO4 < 5.~	50	0.0240
" + DRC.	42 (51)	0.0219 (0.0234)
Dolomite < 5,4	45	0.0266
" " + DRC	. 41 (48)	0.0318 (0.0247)
CaCO ₃ < 5,4	39	0.0301
" " + DRC •	45 (45)	0.0297 (0.0213)

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respectively, and indicate no unusual settling characteristics.

(c) Conclusions

1. The effect of mixing certain mineral dusts of > 5 micron particle size with silica of average particle size <u>ca</u>. 1 micron is to produce mixtures which settle more slowly than would be expected from a consideration of the settling characteristics of their components.

This effect occurs also when 5-10 micron silica is substituted for the mineral dust in the mixtures, 5-10 micron silica having a particle size comparable with those of the minerals.

It is clear, therefore, that the effect is due, not to peculiarities inherently associated with the chemical constitution of the additive compound, but rather to effects caused by the alteration in the particle size distribution of the dusts.

2. Mixtures of these mineral dusts of particle size < 5microns with silica of <u>ca</u>. 1 micron average particle size, settle at rates intermediate between those for their components. The experiments carried out have not confirmed reports which state that these minerals increase the rate of settling of their mixtures with silica over the rates for the separate components.

SECTION 4

The Rapid Determination of Specific Surface using the Dust Apparatus

(a) Introduction

The specific surface of a powder or dust is the summation of the areas of all the particles contained in unit weight of a representative sample, usually expressed in square continetres per gramme.

A knowledge of the specific surface of a dust is considered of great importance in the study of silicosis and pneumokonicsis as it is related to rate of solution, absorbtion and sedimentation etc., and therefore an examination of the properties of a range of dusts must include the determination of their specific surfaces which give single figures representative of the overall fineness of the appropriate dusts.

The optical methods of specific surface measurement usually depend on the estimation of the light cut off by a homogeneous suspension of the dust in a liquid when a parallel beam of light is passed through it. It has been shown that the mean projected area of a particle of any shape when in turbulent suspension or random motion is equal to one quarter of the geometric surface of the particle (40). Heywood (52), has evolved the following equation for the estimation of specific surface by this method:

$$I_a = I_o e^{-SCX/4}$$

where, $I_a =$ intensity of the light transmitted through the (suspension)

 $I_o =$ intensity of the light transmitted through the C = concentration of dust (g./c.c.) (pure liquid S = specific surface (sq. cm./g.)

X =length of light beam (cm.).

The equation, however, breaks down for very small particles approaching in size the wavelength of the light used (41, 42). For these particles (there is conflicting evidence available as to their exact size, but it is thought to be about 1 micron), extinction of light becomes less than would be expected from a simple geometric consideration of the particles. This fact has already been demonstrated in Section 2. and is an inherent drawback, leading to underestimation of the specific surfaces of dusts containing large numbers of particles of minute size.

The method of deriving results with the Dust Apparatus nullifies this objection as the initial galvanometer reading becomes immaterial, only the relationship of the others to this initial reading being of importance when the figures are converted to percentages, and the results depend only on the overall rate of sedimentation of the particles.

Previous experiments suggested that the Settling Factors as obtained with the Dust Apparatus were approximately proportional to the specific surfaces of the dusts they represented, and it appeared

that it might be possible, by calibrating the apparatus using dusts of known specific surface to produce a curve; Settling Factor \ll Specific Surface, which would enable the apparatus to be used for the rapid determination of specific surface.

(b) Experimental

1. Calibration of the Apparatus and Initial Results

Since the apparatus had been fitted with new photo-cells the settling curves for the graded dusts were repeated using 0.05 g. material. Settling Factors obtained from these curves are shown in Table 17 and the calibration curve, obtained by plotting the Settling Factors against specific surface, in Graph 31.

Thirteen dusts of various specific surfaces were then made up by mixing known weights of the graded dusts, their specific surfaces calculated from the weights used, followed by the determination of their Settling Factors from the settling curves obtained using the apparatus. By means of the Settling Factors it was then possible to read off from the calibration curve, figures for the specific surfaces for comparison with the calculated values.

The results obtained are given in Table 18.

It may be observed that very good agreement is usually obtained between calculated and experimental values. Three results, however, and possibly four, appear to be anomalous giving excessively high experimental results. These are Nos. 6, 10 and 12 and possibly No.3, whose experimental values are between 25 and > 100% higher than

Sec. Sec.

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	Repeat	Figures	for Grade	d Dusts						
	(new photo-cells)									
				· .						
•	5-10 ₁₁	3-5 м	1-3 , 1	DRC.	<1, m					
Settling Factor	4.7	20.3	38.0	53.6	60•3					
Specific Surface (sq.cm./g.)	3,400	6,000	10,400	20,700	29 , 800					
2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2										
1. 										



Calculated and Experimental Specific Surfaces of Mixed Dusts

No.	% Composition by Weight	Specific Surf.(calc.)	Settling Factor	Specific Surf.(exptl.)	% E1	ror
1	DRC. 75: 1-3,4 25	18,125	51	18,200	0,4	high
3	DRC. 75: 3-5 ~ 25	17,025	54	21,400	25.6	11
5	DRC. 75: 5-10, 25	16,425	47	15,200	7.4	low
2	DRC. 25: 1-3,4 75	12,975	44	13,200	1.7	high
11	DRC. 1-3: 3-5: 5-10x 25 each	10,175	38	10,400	2.2	11
4	DRC. 25: 3-5,4 75	9,675	35	9,500	1.8	low
7	1-3, 75: 3-5, 25	9,300	31	8,500	7.1	14
9	1-3,4 75: 5-10,4 25	8,700	32	8,700	0	•
6	DRC. 25: 5-10,4 75	7,875	50	17,500 >	100	high
8	1-3~ 25: 3-5~ 75	7,100	24	6,800	4.2	low
13	DRC. 3: 3-5/4 97	6,460	21	6,100	5.6	78
10	1-3, 25: 5-10,475	5 ,30 0	31	8,400 >	6 0	high
12	DRC. 3: 5-10,4 97	4,135	35	9,500 >	100	11

calculated.

The experimental values are obtained from the Settling Factors determined from the smooth curves drawn through points obtained by averaging percentage galvanometer readings from duplicate experiments. Hence, the Settling Factor obtained in any instance is not necessarily the average of the Settling Factors of the duplicate experiments.

2. The Investigation of Anomalous Results

It may be observed that Nos. 6, 10 and 12 were made up of DRC. 25% + 5-10 micron 75%, 1-3 micron 25% + 5-10 micron 75%, DRC. 3% + 5-10 micron 97%, respectively. That is, each mixture contained a small weight of fine particles combined with a larger weight of coarser material. The only difference between these mixtures and those which gave satisfactory determinations of specific surface is in particle size distribution.

The anomalous results make it appear that the small particles in these dusts had an excessive effect in the sedimentation out of proportion to their weight. This effect is manifested in a slower rate of sedimentation and hence the high specific surface, since, in the method of estimation, rate of sedimentation is linked with specific surface. The phenomenon has also been noted in the mixing of mineral dusts > 5 microns in size with DRC.

Additional mixtures when made up gave similar results when the size range was wide and when the weight of small particles was half or
less than half the total weight of the mixture. This is illustrated in Table 19.

It may be observed from the figures in Table 19. that Nos. 14 and 18 give comparable calculated and experimental values; 4.2% low and 3.9% high respectively, and while their size range is wide the percentage of small particles by weight is large. Nos. 15, 16 and 17, and 16 in particular, give, on the other hand, excessively high figures. While their size range is also wide the percentage of small particles in 16 is small (3%) and in 15 and 17, 50%. The experimental figures for the latter two dusts are closer to the calculated values than is that for No. 16.

From the results quoted it is interesting to compare graphs of the variation in calculated specific surface with percentage composition with those of the variation in experimental specific surface with percentage composition by weight of the constituents of the mixtures.

The curves for DRC., + 5-10 micron, 1-3 + 5-10 micron and 1-3 + 3-5 micron mixtures have been drawn in Graphs 32. 33 and 34.

The calculated values give curves of a straight line form when plotted against percentage composition. The curves obtained, however, for the experimental specific surfaces for DRC. + 5-10 micron and 1-3 + 5-10 micron mixtures rise initially, for small percentages of the smaller constituent, greatly above the calculated values. They eventually approach the straight line somewhere beyond 50% of the smaller constituent. The experimental curve for the 1-3 + 3-5

Table 19

No.	% Composite by West	sition ight	Spe Sur	cific f.(calc.)	Settling Factor	Specific Surf.(expt ¹ .)	%] -	Error
14	DRC. 90:	مبر10-5	10	18,990	51	18,200	4.2	low
15	DRC. 50:	5-10,4	50	12,150	45	14,000	15.3	high
16	1-3 <i>1</i> 3:	5 - 10,u	97	3,813	20	5,900	55.0	11 12
17	1-3µ 50:	5 - 10µ	50	7,000	33	8,900	27.7	11
18	1-34 90:	5 - 10 <i>u</i>	10	9 , 720	37	10,100	3.9	17





micron mixture is, within experimental error, close to the calculated curve.

The divergence between experimental and calculated curves is much greater for the mixtures containing DRC., than for those containing 1-3 micron silica, and it appears, therefore, that the wider the size distribution the wider the divergence.

This point is further illustrated from the results obtained from the examination of several commercial silica dusts, and also of two mine dusts. Particle size analyses were carried out using the Andreasen Pipette (Section 6.) and are tabulated in Table 20. It should be noted that these dusts were not originally of an airborne nature.

Specific surface measurements were also made as previously using the Lea and Nurse method and the figures obtained are shown in Table 21. with comparative results obtained with the Dust Apparatus.

Even greater discrepancies are shown than for the smaller synthetically prepared dusts and this links up with the again greater range of particles, which extends from <1 micron to >100 microns. A further reason for the wider discrepancy is the fact that these dusts contain a considerable proportion of material >10 microns in size which is suspended for too short a time to be recorded by the apparatus.

3. The Effect of Particle Size Distribution on Rate of Sedimentation

As has been previously shown, it is possible from the settling curves to obtain figures for the average terminal velocities of the

	Particle	Size	Analyses	of Indus	trial Dus	ts	
Stokes' Diam.(~()	s ₃	SĄ	WC ₄	₩C ₆	Mine Dust 1	Mine Dust 2	3
> 100	19.4	6.1	7.1	16.7	4.3	tanîn seri O	1
20-100	30.2	47.4	53.4	52 .7	33.5	40.9	A
10-20	26.1	22.5	16.6	2.6	37.4	52.0	7
5-10	11.3	11.0	10.0	17.8	13.6	4.2	4
3-5	5.6	6.0	3.3	2.6	6.3	0.7	5/0
1=3	6.0	4.9	7.0	5.5	1.8	0.2	4
<1 (1)	1.4	2.1	2.6	2.1	3.1	2.0	%

Table 20

Table 21

	Comparat	of Industrial Du	cific Surfaces sts	
e e generation e e e	:			
		Air Permeability	Dust Apparatus	
•	S ₃	3,220	20,560	
tij dat de	S4	3,109	23,200	
anti ya s	WC ₄	3, 538	22,000	
e Bac	wc ₆	2,825	29,400	
	Mine Dust 1	5,4 80	25,200	
	Mine Dust 2	4, 489	26,600 (sq.cm./	′g.)

dusts dispersed in the apparatus. When these are calculated for mixtures 1 to 13, the figures set down in Table 22 are obtained. The terminal velocities of the mixed dusts are calculated from the slopes of the curves drawn and annotated in Graphs 35, 36 and 37. Average particle diameters calculated from the terminal velocities are also given.

It may be observed that, as has previously been stated, the average terminal velocities (V_{12}) for the individual graded dusts show that the rates of sedimentation of DRC., and 1-3 micron silica are greater than would be calculated from the average particle diameters obtained from their true specific surfaces. From V_{12} , the average particle diameters are 1.58 and 2.23 microns as against actual values of 1.09 and 2.16 microns respectively. This difference would, in actual fact, probably be even greater but for obscuration by turbulence in the chamber. The inference is that both these dusts exhibit a considerable degree of aggregation when dispersed in the chamber, the aggregation being relatively greater for the smaller dust (DRC.).

3-5 and 5-10 micron silica, on the other hand, give average terminal velocities which are considerably less than calculable from Stokes' Law. Average diameters of 2.63 and 3.88 microns as against actual values of 3.76 and 6.46 microns respectively are produced, indicating an overall slower rate of sedimentation. The inference is that aggregation is considerably less, if it exists, and is

Table 22

Terminal Velocities of Mixed Dusts

	Av.Particle Diam.calc. from Speci- fic Surface (M)	T.V. calg. from Stokes' Law (cm./sec.)	T.V. from Graph ^V 12	Av. Particle Diameter appropriate to V ₁₂
DRC .	1.09	0.0108	0.0227	1.58
1-3,u	2.16	0.0402	0.0427	2.23
3 - 5,u	3.76	0.1171	0.0572	2.63
ىر10-5	6 •46	0.3414	0.1210	3.88
1	1.25	0.0145	0.0178	1.39
3	1.34	0.0166	0.0187	1.43
5	1.38	0.0176	0.0269	1.70
2	1.74	0.0260	0.0244	1.69
11	2.23	0.0428	0.0414	2.19
4	2.34	0.0471	0.0240	1.61
7	2.43	0.0493	0.0439	2.27
9	2. 6 2	0.0579	0.0425	2.24
6	2.87	0.0690	0.0206	1.57
8	3.19	0.0858	0.0464	2.34
13	3.51	0.1028	0.0447	2.06
10	3.80	0.1196	0.0324	1.98
12	5.48	0.2460	0.0152	1.36





certainly not great enough to obscure the retarding effect of turbulence.

An examination of the figures for the mixed dusts, however, shows that: 1. Nos. 1, 3 and 5 have average terminal velocities slightly greater than calculable from Stokes' Law, and these dusts contain 75% of DRC.

2. Nos. 2, 11 and 4 contain 25% of DRC., and give slightly lower terminal velocities than calculable. No.13 contains 3% of DRC.

3. Nos. 7, 8 and 9 have slightly lower terminal velocities and contain either 25 or 75% of 1-3 micron silica.

1., Suggests that the large amount of DRC. is the overriding factor and aggregation still occurs sufficient to mask the effect of turbulence.

2., Suggests that the large amount of constituent other than DRC. is the overriding factor and insufficient aggregation occurs to mask the effect of turbulence.

3., Suggests that the aggregation of the 1-3 micron silica is masked by the larger material so that the composite dust shows to a slight degree the effect of turbulence.

Dust No. 11 contained a graded range of particles and the terminal velocities (experimental and calculated) are very close together, suggesting that the aggregation balances the effect of turbulence.

Dusts Nos. 6,10 and 12, on the other hand, have experimental terminal velocities very much lower than calculated and this suggests that the small amounts of fine particles have been considerably de-aggregated. This, coupled with the effect of turbulence, suffices to reduce the overall rate of sedimentation by a large amount.

Thus, recapitulating:

No.	T.V. calculated. (cm./sec.)	Particle Diameter (4)	T.V. experi- mental.	Particle Diameter
6	0.0690	2.87	0.0206	1.57
10	0.1196	3.80	0.0324	1,98
12	0.2460	5.48	0.0152	1.36

These dusts give anomalously high specific surface determinations.

It would appear, therefore, that there may be considerable variation in the dust producing power of finely divided materials as measured by their terminal velocities and this power may or may not be measured by specific surface determined by standard methods (e.g. air permeability). Hitherto it has been generally accepted by workers in this field that, exclusive of other considerations such as composition, specific surface, and in particular, the surface of the particles <5 microns contained in a dust, gives a reasonable estimation of the danger likely to be associated with the dust. These experiments show that the presence of much large material in a dust containing very fine particles may modify considerably the overall rate of sedimentation of the dust. It is apparent that the more slowly a dust settles the longer is it available for inhalation and is therefore potentially more dangerous.

(c) <u>Discussion</u>

Two main reasons are thought to explain the phenomenon related above:

1. The inclusion of large particles (10 microns) in excess weight (not necessarily excess numbers) in a dust containing a proportion of very small ones (1 micron) increases the inherent dispersibility of the small ones. That is, a small closely graded group of particles has a dispersibility inherent to it, and probably depending on electrostatic forces developed among the particles, so that, on dispersion, it disperses in aggregates, the aggregation being greater as the particle size decreases. Proof of this inherent dispersibility peculiar to each dust, has been in evidence throughout the work with the Dust Apparatus, which has given constant percentage settling curves for each separate dust.

The alteration of the particle size distribution of a dust by the inclusion of sufficient particles of widely differing particle size alters the inherent dispersibility and causes greater dispersion of the small particles to an extent which, in many cases, obscures the rapidity of the sedimentation of the larger particles. The average terminal velocity is therefore less than would be expected by calculation from the now greatly reduced specific surface

measured by air permeability.

Since specific surface as determined with the Dust Apparatus depends on rate of sedimentation, when this occurs an overestimation of the specific surface is obtained.

2. A further, and, it is considered, lesser cause of the effect, is thought to be increased turbulence in the chamber caused by the settling of the larger material affecting the settling of the small particles. Evidence for this is obtained from a consideration of the properties of Mixture 12. This mixture is made up of DRC. 3% + 5-10 micron silica 97% and gives an experimental terminal velocity far below the calculated value. Values for N_{+} (the number of particles settled up to time t) were calculated for the mixed dust from the known figures for percentage composition and specific surface of the constituents. These values were then converted to comparative 1-3 micron numbers [Section 2, 2(b)] from which were subtracted the figures for N_t for 5-lQusilica alone. Values were therefore obtained of N_t for DRC., as affected by the addition of 5-10 micron silica, assuming that the sedimentation of the 5-10 micron material is unaffected by the presence of DRC. It may be observed from Graph 38 that the effect appears of considerable magnitude and is of a fluctuating nature. Graph 38 shows Nt values drawn proportional to time for DRC. as affected by 5-10 micron silica, compared with those for DRC. alone.

The limitations of this evidence are realised by it is included (ut/



to illustrate the complexity of factors which may affect the sedimentation of dusts in air.

(d) Conclusions

The Dust Apparatus has proved effective in estimating the specific surfaces of a limited range of dusts, namely, those whose particles are under about 10 microns and are within a limiting range of about 5 microns.

Dusts above 10 microns settle too fast for measurement in the apparatus, and dusts with a wide range of particles give erron@ously high values due to factors affecting the sedimentation of the dusts which depend on the particle size distribution.

The accuracy of the estimation depends on the specific surface of the dusts but is usually better than 10% for those dusts within the range specified above.

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SECTION 5

The Evaluation of 'Health Hazard'

Acceptance of the solubility theory of silicosis stresses the importance of the surface areas of dusts of similar composition as a measure of their pathogenicity, and since the upper limiting size of 5 microns is generally accepted, the surface area of . particles below this size in a dust is of even greater significance. The most recent attempt (28), to obtain a suitable comparative measure of the health hazard to be associated with dusts, aims at obtaining as much intermediate information as possible (e.g., mass concentration of total airborne dust, specific surface of total dust, mass concentration of the dust below 5 microns and a comparative measure of the surface area of the dust below 5 microns); but a composite figure is also obtained which is defined as the surface area of the particles below 5 microns, expressed as a weight percentage of the particles below 5 microns contained in a "normal dust". "Normal dusts" being defined again, for convenience, as dusts whose specific surfaces are approximately proportional to the weight percentage of particles below 5 microns which they contain.

After collection of the sample by the gravimetric method, the dusts are evaluated in the laboratory by means of a photoelectric light extinction method, the dust being dispersed in alcohol. Several estimations were originally carried out, using different liquids, to determine the most suitable, i.e., giving most complete dispersion. Thus, of three dusts (53), the following values for

dispersing power as measured by variation in specific surface in different liquids shown in Table 23 were obtained. Specific surface may be used as a comparative measure of dispersion as, if there is aggregation or flocculation of the particles, the obstruction of the light beam will be erroniously low, resulting in an erroniously low value for specific surface.

The results quoted show that dispersion of the dust depends on the liquid used and, according to other figures obtained by the same workers is also constant for a particular dust in a particular liquid. This tends to confirm previous remarks (Section 4) regarding the inherent dispersibility of particular dusts with air as dispersing medium.

Thus, it is apparent that when maximum dispersion of the dust is aimed at and achieved, using alcohol, it does not necessarily represent the original dispersion of the dust in the air, or even the subsequent dispersion if the dust is resuspended inair. It appears that while the South Wales method or any specific surface method will give, within experimental error, the maximum external surface of the dust particles, it does not necessarily bear an exact relationship to the surface of the dust which, depending on its dispersion, will find its way into and be trapped by the lung.

Two factors are involved. If a dust is dispersed in an aggregated form it will settle more rapidly than it would if it were not aggregated, and less will hence be available for inhalation.

Table 23

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na sere do trado deservo. Nacional	熟识意识的(含)。Quana编成(1)

The Specific Surfaces of Dusts Dispersed in Various Liquids

Liquid	1	Dust 2	n a la transmissión de la compañía. 3
		a de la composition de	
Water	5,090	7,240	6,350 ⁷⁶⁸ • 1,564 • 1,56
Water + 1% Perminal	7,560	8 , 700	en on oberg of generalization – en le le ign to recteur 25
Alcohol	8,260	10,500	12,000 periiske is.
Alcohol/water 50/50	8,070	10 , 300	11,500 11,500 Ally Related dat of
Petroleum ether	14 ,730	6 , 430	- s (sq.cm./g.)
segtering in the first state.	haptens.		anna havovan that discourse
ster of the state shou	ld be this	n. inter	mocelli in algebrationstic
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If the dust is aggregated it will depend on the size of the aggregates whether it is eventually retained in the lung or expired. As Van Wijk and Patterson state (13), there is a natural tendency for the larger particles to deposit in the large air passages of the lungs and for the smaller ones to deposit in the smaller passages. The large particles are therefore more easily expelled by the physiological dust expulsion mechanism of the lungs. It is conceivable that this will apply to large aggregates. The opposite might be true however of small aggregates. Removal of particles below 0.2 microns from the atmosphere by the lungs is under 25% according to Van Wijk and Patterson, but if these particles are aggregated, then the aggregates larger than 0.2 microns will be removed in greater proportion (13). When finally ingested these aggregates will probably act as single particles. It is obvious, therefore, that the mechanism is too complicated to say with certainty exactly what happens. It appears, however, that dispersion of the dusts should be taken into account in any estimation of health hazard.

Since it has been shown (Section 4) that the dispersion of a dust depends on its particle size distribution, it will therefore also determine the amount of dust which will be suspended in the atmosphere by agitation of the dust already deposited on roadways etc., and raised by the removal of coal and rock.

When the figures for health hazard, appropriate to dusts of

known specific surface, by the South Wales method are compared with Settling Factors for the same specific surfaces read off from the calibration graph opposite page 71, the results shown in Table 24 are obtained.

It may be observed that for dusts dispersing "normally" the Settling Factors place them in the same order and indeed above 4,000 sq.cm./g. specific surface, with figures almost identical to those obtained with the South Wales method. The Settling Factors for specific surfaces under 4,000 are apparently unduly low but this is not a disadvantage as 3,000 represents an average particle diameter of <u>ca</u>. 7.5 microns; particles which are thought to be relatively unimportant from the point of view of health hazard. The dusts which give high Settling Factors owing to unduly high air-dispersion would not give high figures for the health hazard with the South Wales method which is directly linked with specific surface determined by the light extinction method.

It is considered, therefore, that the Settling Factors give a better indication of health hazard; as, for normal dusts, the results are parallel to those of the South Wales method, but for a dust with abnormally high dispersion the Settling Factor records the fact with a high value and therefore indicates the greater hazard to be met from increased possibility of inhalation.

Line - Alexandri I<mark>Table 24</mark>7 an<u>ne Berli Alexa Bork</u> 14 - Partezie El Conteste - Mainten Alexandri Alexandri

Comparison of South Wales and Dust Apparatus Figures

Specific Surface (sq.cm./g.)	South Wa Metho	ales Settling with od Factor	
etiste en operation d'a 3,000 Exampetano de la arrigaiscerte	12	teches as abing of seadar O 1991 - Teches and totas renew	•
4,000 - 1 - 4,000 - 1 - 4	15	en en groban u s eg - Porthermo t	
wayo terta, walao bala dawa landa ing 6,000 dewa terta a sa sa sa sa sa sa sa sa sa	21	a you yoof eenil doos not give 20 harmadi aan while this off	× .
(A.14) - 8,000 - 10,000 - 10	26	a and the second 29 reasonations	n J
ີ 18 ເປັນ 2000 ເຊຍ ເຊຍ ໃນ 1 2,000 ເປັນມີເຫຍັດໃຫ້ເຮັດ ອ້າກອິດ ຫຍັດ ແລະ ເອດ	37		
16.000 (10.00) (10.00)	48	48	

(1) and all laisan tensopretsions in some tensor of the source of a like string one of adaman all laisan tensors on a source of the source of a like tensors and the tensor the constraint and like and a source of the line of the source of a like tensors and the tensor of like tensors tensors of the source of the line of the tensor and the tensors and the constraint tensors tensors of the source of the source of the tensors and the tensors of the tensors tensors of the source of the source of the tensors of the tensors and the constraint tensors of the source of the source of the tensors of the tensors of the constraint tensors of the source of the source of the tensors of the tensors of the constraint tensors of the source of the source of the tensors of the tensors of tensors of tensors of the source of the source of the tensors of the tensors of the tensors of tensors of tensors of the tensors of the source of the tensors of the tensors of the tensors of tensors of tensors of the tensors of tensors of tensors of the tensors of the tensors of tensors of

SECTION 6

General Discussion and Suggestions for Future Work

1. Photoelectric Dust Sedimentation Apparatus

It is proposed to suggest modifications which, in the light of experience, would improve the accuracy of the apparatus.

The photo-cells are of the selenium layer type and with persistent use, fatigue occurs. The effect of this has been minimised by switching on the lamp only during the taking of readings. Long-term fatigue, however, also occurs and necessitates renewal of the cells after about six months of continuous use. Furthermore, according to some workers (53), this type of cell does not give an entirely linear response to light intensity and while this effect is small and is considered negligible in the present circumstances, it is a basic defect. It is therefore worth considering the use of valve type photo-cells such as those produced by Radio Electronics Ltd., Merton Road, London.

In spite of the precaution of intermittent use of the lamp, some heating occurs which causes excessive turbulence in the chamber. It is suggested that in any future apparatus a heat filter should be incorporated in the lamp housing. A transparent container filled with water would be ideal but would necessitate rearrangement of the lamp to keep the lens in focus. Since in the present apparatus the lamp is fixed, it would not be possible to make the necessary adjustment without extensive alteration. Also to reduce turbulence,

the chamber could be thermally insulated from the atmosphere. Terminal velocity determined under these conditions would then give a more accurate picture of the aggregation of the particles suspended in the chamber as there would then be less reduction in velocity owing to turbulence.

The apparatus described was intended for use exclusively in the laboratory. It is thought, however, that the arrangement provides a convenient basis for the development of an apparatus for use underground, which, if coupled to a recording device, would enable a continuous picture of underground dust concentrations to be obtained. The arrangement envisaged is basically similar to the present apparatus, but incorporating, instead of the dust chamber, a box, open at top and bottom through which the mine atmosphere would circulate freely. The dimensions of the box would have to be determined by experiment and would probably be considerably greater than those of the present chamber. It would be necessary also to house the photo-cells in cylinders of a gas-tight nature. In order to make the apparatus portable it would be necessary to actuate the lamp by battery.

It would, of course, be impossible to differentiate between concentration and size of particle by this arrangement but a useful record of the fluctuation of dust conditions would be produced, thereby showing immediately when certain fixed standard were exceeded.

2. The Suppression of Airborne Dust

(a) Mineral Dusts

It has been shown that mineral dusts of the type examined are useless for the suppression of airborne silica, whether the minerals are less than 5 or greater than 5 microns insize, and indeed, the > 5 micron material increases the dispersion of admixed finely powdered silica. This point suggests that, owing to the likely increased suspension in the atmosphere of dangerous material, it would be unwise to use minerals greater than 5 microns in size for dusting in mines. Stone (mineral) dust is used in mining for the main purpose of diluting the airborne concentration of coal dust and thereby decreasing the possibility of the propagation of explosions. > 5 micron mineral dust would therefore be expected, when mixed with fine coal and subsequently disturbed, to allow of the suspension in the mine atmosphere of more discrete and probably finer coal particles.

Two factors are involved. Firstly, to reduce the danger of lung disease, aslittle dust as possible should be suspended in the atmosphere. Secondly, to reduce the danger of explosion, any dust in the atmosphere should contain a certain additional proportion of incombustible dust. These requirements are mutually conflicting.

The use of < 5 micron mineral would not increase the dispersion of its mixture with a similar size of other material but would still be efficatious in the prevention of explosions.

Skinner and Graham (54), express concern at the high proportion of particles less than 5 microns in material used for dusting in mines, as they consider that it is unwise from a medical aspect to overburden the atmosphere unduly with particles of a size believed to be a danger in the production of lung disease. This is particularly so when it is observed that many of the stone dusts which they examined contained as much as 40% of free silica. These dusts were mainly produced from shales. The limestones, gypsum and precipitated calcium carbonate which they mention contained a very much lower proportion (mostly under 1%) of free silica.

The opinion of the author is, therefore, that for dusting in coal mines the stone dust should be as fine as possible, but if economically feasible, those materials containing a high proportion of free silica should be replaced.

(b) Spraying

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The experiments have shown that the most effective reduction of airborne silica dust is likely to be obtained using sprays of wetting solutions. From the results obtained with water sprays it is concluded that, within the range of spray sizes used, the most effective average droplet size is approximately 30 microns. Whether droplets smaller than this size would be more effective should be determined. It is suggested, also, that to obtain maximum removal of airborne dust the greatest quantity and intensity of spray compatible with safety and confort under the prevailing conditions

is required. Sprays should also be ejected from the roof vertically downwards to make use of the inherent gravitational direction of the dust and to enhance the sweeping action of the spray droplets.

Further work should be carried out on suspended silica dust of size less than 1 micron and on other materials of varying size (e.g. coal) to determine the effect of spraying. Further data are also required on the effect of concentration of suspended dust on its reduction by spraying.

3. The Measurement of Specific Surface and the Health Hazard

The apparatus provides a convenient and rapid means for the measurement of the specific surface of small amounts of dust below 5 to 10 microns in size but has the disadvantage that dusts having a small percentage of fine particles coupled with a wide range of particles are seriously overestimated. For the estimation of health hazard, however, this fact includes high up in the assessment of the relative danger of a range of dusts, those dusts which show a slow rate of settling owing to more complete dispersion.

The original calibration of the apparatus, based as it is, on the sedimentation of closely ranged dusts is seen, therefore, at high specific surfaces, to be artificial. It is to be expected that if the apparatus were calibrated using dusts of known specific surface but having widely ranged sizes, the specific surfaces of closely ranged dusts such as DRC., would, owing to aggregation, be underestimated.

The study of dispersion would provide an interesting extension of the present work. It has been observed by many workers that as a group of particles is reduced in size, so does its ability to disperse decease. It has not, however, been previously stressed that the inclusion of larger particles in a group of very small ones would considerably increase the time of suspension in the atmosphere. The explanation of the effect is probably bound up with the electrostatic properties of the particles and, if so, is additional evidence for the theory advanced by Boning (55), that electrostatic charges developed within a group of small particles derive from their mutual collision and depend on their sizes. A profitable future line of research , therefore, would be the study of the electrostatic properties of graded silica and this might develop into a major study of fundamental significance.

Additional advantages of the Dust Apparatus for evaluating the health hazard are enumerated below.

1. Great rapidity. A settling curve may be obtained in approximately 30 minutes. The apparatus therefore lends itself to routine work.

2. 0.05 g. of dust is sufficient.

3. Independence of the decreased light extinction of particles less than 1 micron in size.

4. Sedimentation in air rather than in a liquid is a closer simulation of practical mining conditions.

SECTION 7

Experimental Methods of Particle Size Determination

in the Sub-Sieve Range

Three established methods of particle size measurement, based on different principles, have been made use of in this thesis. They were chosen with due regard to the purpose for which they were required. Two of these methods give the particle size distribution of a collection of small particles and the other an estimate of the overall fineness of specific surface of the material. It is possible, if necessary, to calculate the latter from the former but not vice versa.

No attempt is made here to review methods of particle measurement which would be outside the scope of this work. The purpose of this section is to record the experimental techniques adopted.

The three methods were as follows:

Specific surface: Lea and Nurse, Air Permeability.

Particle size distribution: Andreasen Pipette,

Microscope.

1. Lea and Nurse Air Permeability Method (33)

(a) Introduction

The basis of this method is the measurement of the rate of flow of air through a bed of a known weight of the material, packed to a fixed depth in a cylinder of constant dimensions. The apparatus used may be seen in Figs. 7 and 8, which are self-explanatory.

· PERMEABILITY APPARATUS ·





A measure of the specific surfaces of various grades of powdered silica and other materials was required for checking the size grading of the particles and for the correlation of results obtained with the Dust Apparatus. It is stressed that the figures obtained using this method are not absolute, but are individually comparative, and comparable also with values obtainable by sedimentation methods (56).

Specific surface is calculated from the following equation:

$$s = \frac{14}{\sigma(1-\epsilon)} \sqrt{\frac{\epsilon^3 Ah_1}{CLh_2}} \dots \dots \dots$$

here, $D = \text{specific surface (sq.cm./)}$	ere,	q.cm./g.
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density of material $(g_{\bullet}/c.c.)$ 9 -

porosity of packed bed = volume of pore space/ € ≈ total volume of bed

cross-sectional area of bed (sg.cm.) [5.289 sq.cm.] A =constant of flowmeter (c.g.s.) $[3.951 \times 10^{-6} \text{ c.g.s.}]$ C ≈ depth of bed (cm.) [1.011 cm.] L = difference in level of manometer (cm.) $h_1 =$

 $h_2 =$ " flowmeter (")

The figures in the large brackets give values of the constants for the apparatus used.

 $\boldsymbol{\xi}$, the porosity, is calculated from the density of the powder and the volume filled by it in the following manner:

 $\leq =$ volume of pore space/total volume of bed

= volume of pore space/5.348

wł

volume of pore space = total volume of bed - real volume of powder

= 5.348 - weight/density.

 $\therefore \leq = \frac{5.348 - W/D}{5.348}$

and.

A factor not taken into account in the specific surface results on graded silica which follow is the slight decrease in density of the silica (quartz) which occurs during prolonged grinding and is thought to be due to the formation of a crypto-crystalline layer on the surface of the particles (57, 58). However, allowing for a decrease in density of 0.05; i.e., from 2.65 to 2.60 g./c.c., the error in specific surface would, in any event, be less than 2% and is considered small enough to be neglected.

Theoretically, values for specific surface calculated from equation 1. should be independent of the value of \lesssim . In actual fact, however, there is some variation depending on the value of \lesssim used. This variation is discussed at length by Lea and Nurse in their later publication (56), and also by Keyes (59), who has developed a correction of the original equation based on his own results.

The procedure therefore adopted was to carry out all determinations using a constant value for the porosity function. This value is obtained from the approximate average of the weights of the largest and smallest sizes of the powders whose specific surfaces it is proposed to estimate, which, when compressed in the

permeability cell, give a suitably packed bed.

The porosity must be carefully chosen when a range of powders is to be examined, because a porosity suitable say for 5-10 micron silica might require a weight which if used of < 1 micron silica would have a volume which would be impossible to compress into the permeability cell. Conversely, a suitable weight of < 1 micron silica might be such that if taken of 5-10 micron silica would have a volume which would not completely fill the cell. The porosity most suitable must therefore be found from a weight of material determined by trial and error.

(b) Experimental

The densities of the powders were determined using a 25 c.c. pyknometer with distilled water (ethanol for water soluble dusts) as displacement liquid, and evacuating the bottles to displace air from the small particles.

A weight was then chosen which gave a suitable bed of both < 1and 5-10 micron silica, and the porosity calculated. This porosity, used throughout, had the value 0.5873.

For powders of different densities, a factor was calculated which when multiplied by the appropriate density gave the weight of the particular powder to be compressed into the cell to give the above porosity: for example:-

> $\leq = 0.5873$ total volume of bed = 5.348 c.c. $\therefore 0.5873 =$ volume of pore space/5.348

volume of pore space = 0.5873 x 5.348 = 3.139 c.c.
Real volume of powder = Total volume of bed - Volume of pore
space

= 5.348 - 3.139 = 2.209 c.c.

Weight required = 2.209 x or (density of powder)

The appropriate weight of powder, previously dried in vacuum, was weighed into the permeability cell containing a No.1. Whatman filter, and compressed carefully with the plunger. The plunger was then withdrawn slowly with a slight initial rotary motion, the cell connected to the apparatus and dry air passed through it. Readings of h_1 and h_2 were taken for five different rates of air flow, and the average values used in the specific surface equation 1. Duplicate determinations were carried out, and the results obtained may be seen in Table 25.

2. Andreasen Pipette Method (60)

(a) Introduction

A measure of the relative weight proportions of fine and large particles in certain ungraded commercial powders was required to confirm that anomalous results obtained with the Dust Apparatus were due to the wide size distribution and relatively large amount of large particles contained in the powders. Since a comparatively large quantity of these powders was available the Andreasen Pipette method was considered as most suitable.

(b) Apparatus

The apparatus consists (Fig. 9) of a vessel of ca. 600 c.c.

Table 25

Porosity 0.5873 Density Weight Mean % Variation Specific Material from Mean $(g_{\bullet}/c_{\bullet}c_{\bullet})$ (g.) Surface (sq.cm./g.) 29,360 سر1 > 2.65 5.8475 29,805 1.5 silica 30,250 0.9 ^x(11,300) tt 11 10,260 1-3~ 10,355 10,450 11 Ħ 5,903 1.3 ^x(5,600) 3-5,1 5,979 6,056 tt tt 3,554 5-10~ 5.4 ^x(3,000) 3,371 3,188 20,810 DRC . 2.61 5.7566 20,730 0.4 20,650 CaS04 > 5~ 2.96 2,617 6.5386 2,624 0.3 2,631 11 ac 5 م Ħ n 17,030 16,980 0.3 16,930 CaCO3 > 5~ 2,724 2.80 6.1852 2,728 0.2 2,733 2.85 Dolomite 6.2956 933 903 3.3 >5~ 873 "< 5 م n u 13,570 13,675 1.1 13,780 2.64 5.8384 3,228 **S**3 3,220 0.2 3,213 11 11 3,170 S_4 3,109 2.0 3,049 WC_4 3,617 2.65 5.8475 3,538 2.3 3,459

continued over page/
Material	Density	Weight	Specific Surface	Mean	% Variation from Mean
WC ₆	2.65	5,8475	2,865 2,784	2,824	1.5
Mine Dust l	2 .73	6.0060	5,486 5,473	5,479	0.1
"2	2.75	6 •0680	4,562 4,414	4 ,4 88	1.7

S₃, S₄ etc. are trade symbols for commercial dust samples supplied by Messrs. Colin Stewart, Winsford, Cheshire.

x The figures in brackets are the values obtained by calculation from the nominal average size of these dusts and show good agreement with the experimental values.

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Table 25 (cont.)



capacity to hold a suspension of the powder in a liquid, calibrated in cm. from a line about 4 cm. above the base to a height of 20 cm. A device for sampling at the zero mark, portions of 10 c.c., is fitted through the neck and is provided with a two-way stop-cock, so that the sample may be withdrawn by suction and discharged into a dish. For very accurate work the apparatus is thermally insulated from the room.

(c) Experimental

The method was modified slightly to suit the requirements.

A weight of the powder to be analysed, sufficient to form a suspension having the solid in proportion of about 1% by volume (ca. 12 g.), was weighed accurately, transferred to the vessel, and made up to the 20 cm. mark with liquid. For silica, water was used but for the mine dusts, ethanol. The vessel was thoroughly shaken to form a uniform suspension. The time at which shaking ceased was noted, and samples withdrawn after times calculated from Stokes' Law (Section 2) equivalent to the settling of all particles in the suspension of sizes over 100, 20, 10, 5, 3 and 1 micron respectively, below the zero line from the level of the top of the suspension. This level decreased by 0.4 cm. for each 10 c.c. sample withdrawn. The samples were transferred to tared porcelain dishes, evaporated to dryness, and reweighed.

The particles are assumed to be uniformly suspended at the start of sedimentation; and, from the initial weight of powder and

the volume of the suspension, the total weight of particles in 10 c.c. may be calculated. Hence, also, the percentage weight of powder still in suspension at the zero level for each sample taken may be determined.

A specimen of the procedure is presented in Table 26.

Thus, a determination is made of the percentage by weight of the powder which is smaller than a known particle size, and from this the percentage weight between-size may be deduced. If a full size distribution is required, it is obtained by plotting percentage undersize against Stokes' diameters and differentiating the resultant curve.

The figures for percentage between-sizes of other dusts are presented in the appropriate place (Section 4, b2).

3. Microscope Method

This was used to obtain a measure of the average size of spray droplets emitted from spraying devices in dust suppression experiments.

The spray droplets were relatively large and measurement was carried out using the low power objective which gave a magnification of approximately times 100. The microscope eyepiece was fitted with a scale previously calibrated in microns from a stage micrometer. One small division of the eyepiece scale was equivalent to 13 microns.

The collection of the spray droplets is described in Section 3, 2b. 1.

Table 26

the include contained for a state 12 g. A total of challed for a solution of challed for a solution of challed for a solution of the s

	Tìr	10 28 (3020) -	Depth (cm.)	Weight i 10 c.c. Sample (n % Sample in Suspension g.)	Stoke Diam	es' % B .(4) S	etween izes
			·		na an a			
25.0	0		-	0.2	100	-	>100	19.4
	25	55.	20	0.1613	80•6	100	28100	20.2
	10m	23s.	19.6	0.1008	50 .4	20	20-100	50 0 20
	4.0m	43a.	19.2	0.0486	5 de la 19 en 1 2 A - 3	١Ó	10-20	26.1
		105.		0.0100	~	TO	5-10	11.3
2h	39m	36s.	18.8	0.0260	13.0	5	3-5	5.6
7 h	13m	50 s .	18,4	0.0148	7•4	3	19	6 0
63h	40m	0s.	18.0	0.0028	the four 1.4	1	T=2	0.0
		Sec	: 	an tain	`		<1	1.4

A suitable field was chosen, and, by rotating the ruled eyepiece, the droplets contained in concentric circles swept out by 10 small division sections of the scale were measured and tabulated; Fig.10. A total of <u>ca.200</u> droplets was measured in widely separated fields, a number sufficient to give reasonably accurate measurement from a collection of uniform spherical droplets. The analysis is, of course, on a numerical basis.

The mean numerical diameter was obtained in the following manner:-

Let n_1 , n_2 , n_3 etc. be the numbers of droplets having diameters of d1, d_2 , d_3 etc.,

then,
$$d_{\text{mean}} = \frac{n_1 d_1 + n_2 d_2 \cdots n_n d_n}{n_1 + n_2 \cdots n_n}$$
$$= \frac{\sum_{n \in d} n}{\sum_{n \in d} n}$$

The results obtained for the four sprays used may be seen in Table 9 (Section 3, 2b, 1.).



APPENDIX 1

Specimen Exa	mple of the Dete:	mination of Tern	ninal Velocity
		DRC .	
Average parti	cle diameter ca	lculated from spe	ecific surface of 20,800
	:	= 1.09 microns	
C _o (i	nitial concentra	ation in particle	es/c.c.) = 2,325,000
		h	= 11.5 cm.
Time (sec.)	% G.R. (settled)	N _t	Log_e (l - N_t/C_oh) -1
10			、
20	6	1,604,250	0.0621
30	10	2,673,750	0.105
60	19	5,080,125	0.211
90	12	3,208,500	0.128
120	12	3,208,500	0.128
180	38	10,160,250	0 .47 7
240	41	10,962,375	0.527
300	46	12,299,250	0.616
360	52	13,904,500	0.733
420	55	14,705,625	0.798
480	61	16,309,875	0.940
540	64	17,112,000	1.021
600	67	20,320,800	1.107

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P.T.0.

Slope = 507/1 = h/V = 11.5/V

(:) V = 11.5/507 = 0.02269 cm./sec. = 1.58 microns.

T.V. of 1.09 micron $= \frac{1.09^2 \times 2.60 \times 981 \times 1.164 \times 10^{-8}}{18 \times 0.001816}$

In which of the sign of the experimentation with any more experimental products are applied by the charger in a point of the second of the second by bits during drilling operations would be an inclusion of possible polymorphic charges which might be about in the crystalline particles of the dusts produced. To a considered that any such charges in the crystalline structure is about of allocais emong drill operators who are exposed to concentrations of airborne dust.

The tests were divided into two sections according to the section according to the sector of the material which was to be drilled, 1. coal and soft ro

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APPENDIX 2

The Temperatures Reached by Drilling Bits in Mining Operations (a) Introduction

This appendix describes experimental work carried out in the initial stages of the research. Since it has no direct connection with the main work, it has been included as an appendix in order to preserve the flow in presentation of the main body of the thesis.

In view of the high surface temperatures which are known to be produced during polishing and grinding processes and having regard to the changes in crystalline structure of the silica lattice which result from heating, it was considered that an estimation of the temperatures reached by bits during drilling operations would give an indication of possible polymorphic changes which might be brought about in the crystalline particles of the dusts produced. It was considered that any such changes in the crystalline structure of airborne silica dust particles might be of gome significance in the causation of silicosis among drill operators who are exposed to heavy concentrations of airborne dust.

(b) Experimental

The tests were divided into two sections according to the hardness of the material which was to be drilled, 1. coal and soft rock, and 2. hard rock.

Rotary drills are used in coal and soft rock, while for brushing operations in hard rock percussive drills are employed.

Temperature Measurement

The methods devised for measuring the temperatures of the bits used in these two drilling procedures were as follows:-

1. Rotary Drilling

A number of rotary bits were prepared each with four 'temperature spots', as shown in Fig.ll. These consist of a hole 3/32" diameter x 1/4" deep near the cutting face of the bit. At the bottom of the hole is a cube of alloy previously prepared and of known melting point, the side measurement of the cube being slightly less than 3/32". On top of the cube is packed a plug of steel wool, the hole being finally closed with a copper plug.

The four 'temperature spots' in each bit contained alloy cubes of different melting points, the four making a series over the temperature range expected to be reached by the bit when in use.

After the prepared bits had been used in a drilling operation the alloy cubes were examined to ascertain which ones had fused. In this way the highest temperature range reached by the bit was determined.

2. Percussive Drilling

The use of 'temperature spots' in the percussive bits was found to be impracticable since the powerful percussive action caused deformation of the alloy cubes by their continuous agitation inside the 'temperature spot' holes. A means was therefore devised to measure the temperature of the percussive bits by a thermocouple.



The percussive drill shanks have a hole <u>ca</u>. 1/4" diameter running throughout their length, which may be used to carry a stream of water to the drill head with the object of laying the dust produced during drilling operations. This procedure is known as 'wet drilling'.

The drills are worked by compressed air and a blast of air directed through the central channel may be used to clear the drill hole at the end of the drilling operation to facilitate withdrawal of the drill shank. The temperatures of the bits were measured during dry drilling so that the results indicate the <u>highest</u> temperatures which the bit was likely to reach in operation.

This central channel offered a most convenient situation for thermocouple leads. The drill, in addition to its percussive action, rotates slowly (20 r.p.m.) during operation, and a method was required to pick up the current from the thermocouple leads since these would rotate with the drill shank.

Details of the device which was designed for this purpose are shown in Fig. 12. The current 'Pick-Up' is held on the drill shank between two steel springs which are kept under compression by two brass clamping rings. The mainbody of the 'Pick-Up' is built of ebonite and consists of two rings (A), fixed to a central core (B), which revolve with the drillshank. A central ebonite portion (C), which bears on the surface of the core (B), remains stationary when the drill shank is revolving. The thermocouple (chromel-alumel)



junction is mounted in a brass bush inside the drilling bit and the ends of the thermocouple are led through the central channel of the shank and are brought outside the shank at a suitable point near the 'Pick-Up' for connection to the terminals set in the ebonite ring (A). The thermocouple potential is thus connected to the copper rings set in (A) and is picked up from these rings by the carbon brushes which are in electrical contact with the copper rings set in the central portion (C). The current is then led from the outer periphery of (C) to the galvancmeter.

The 'Pick-Up' was made by Mr. A. Clunie and Staff (Tech. Chem. Dept. (Workshops), Royal Technical College, Glasgow).

Procedure

Tests using the 'temperature spots' for rotary drilling and the 'Pick-Up' for percussive drilling were carried out at Plean Colliery, Stirlingshire, in conjunction with Mr. D.W.Clelland (Tech. Chem. Dept., Royal Technical College).

Results

The highest temperatures reached by rotary bits in coal (Moh's Hardness, $2\frac{1}{2}$) was found to be <u>ca</u>. 70°C and in soft rock (Moh's Hardness, 2) <u>ca</u>. 100°C.

The percussive drills in the hardest sandston / rock (Moh's Hardness, $6\frac{1}{2}$) reached <u>ca</u>. 120°C.

Conclusions

The temperatures observed were considerably below that required

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for the transformation of quartz to cristobalite and tridymite $(870^{\circ}C)$. It is noted, however, that in percussive drilling of the hardest rocks, that, although the bit as a whole was not raised to a temperature near that required for conversion, incandescent sparks were observed at the start of drilling and it may be assumed, therefore, that extremely small amounts of the rock were being raised to temperatures above $870^{\circ}C$.

It was concluded that no change in the crystalline structure of airborne quartz particles produced in drilling operations could be brought about by the temperatures reached by the drilling bits.

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