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ABSTRACT

Debbi Karen Creed. Development of a Method for Determining the Respirable Mass Fraction of a Bulk Material. (Under the direction of Dr. Parker C. Reist, Ph.D.)

The recent designation of crystalline respirable silica as a carcinogen has created a need for simple laboratory methods to determine the mass fraction of respirable particles in bulk silica-containing materials. Results could influence whether labeling would or would not be required. A liquid sedimentation technique was used to obtain cumulative size distributions for a variety of silica-containing dusts. These results were plotted as cumulative size distributions for the test dusts. The percentage of particles having an equivalent aerodynamic diameter less than 3.5 micrometers was taken to represent the respirable mass fraction of the original bulk material. Validation studies of the sedimentation method were carried out by comparing the results to actual respirable mass fraction measurements. A uniform dust cloud of the test material was generated within an acrylic chamber. Respirable samples and total dust samples (i.e. dust collected on a PVC filter without a precollector) were collected. The ratio of the respirable dust to total dust was used to represent the respirable mass fraction of the airborne material. Side-by-side comparisons of the sedimentation and cyclone respirable fraction measurements were in close agreement with a maximum deviation of $\pm 5\%$. This study concludes that sedimentation

methods can be used for determining the respirable fraction of certain silica-containing dusts. The sedimentation method provides a rapid, inexpensive, and easy method for obtaining accurate and reproducible estimates of the respirable fraction of bulk dust samples.

INTRODUCTION

Inhalation and pulmonary deposition of crystalline silica has long been implicated as the cause of silicosis, a fibrotic disease of the lungs. Recent experimental and human data conclude that there is now sufficient evidence for the carcinogenicity of crystalline silica in rats, and limited evidence for the carcinogenicity of crystalline silica to humans when the silica is inhaled as a respirable dust. These findings were published by the International Agency for Research On Cancer (IARC) in the Volume 42 Monograph on the Evaluation of the Carcinogenic Risk of Chemicals to Humans by Silica and Some Silicates. The IARC findings were incorporated by Occupational and Safety Health Association (OSHA) and added to the National Toxicology Program's (NTP) Sixth Annual Report on Carcinogens.

The designation of crystalline respirable silica as a carcinogen results in federal labeling requirements for products which may generate airborne silica during handling. Materials containing silica are abundant and are frequently used in industrial, occupational and nonoccupational settings. The main source of crystalline, or free silica, is quartz, although it is also found in cristobalite, tridymite, diatomite, and a few other silicate and nonsilicate materials. Silica flour, or silica in its powdered form, is a major component of paints, wood fillers, scouring soaps, and porcelain. Silica sand is necessary for

the production of glass and silica brick, in mortar, and as an abrasive. Industries relying on these processes include glass manufacturing facilities, granite cutting operations, foundries, and operations which involve mining and tunneling in quartz rock (2). Products commonly found in the home which may contain silica include talc, cosmetics, and pesticides. Of late, much attention has been focused on nonoccupational exposures by artists and craftsmen to silica-containing paints, clays, ceramics, and stones.

The federal labeling legislation creates a need for simple laboratory methods to determine the mass fraction of respirable particles in the bulk silica-containing material. These measurements could then be used to determine whether a material does or does not represent an occupational hazard to exposed employees, and consequently, whether labeling would or would not be required. The goal of this research was to develop such a procedure for routine laboratory usage. The method selected for evaluation was liquid sedimentation using the Andreasen sedimentation pipette. Validation studies of the sedimentation technique were carried out by comparing the sedimentation results to respirable fraction measurements taken using 10-mm nylon cyclones.

BACKGROUND

Historically, the microscope has been relied upon for measuring particle size, with particle size being defined in terms of the particle diameter for spherical particles and as Martin's diameter, Feret's diameter, or the projected area diameter for nonspherical ones. Over time, the microscope has given way to more sophisticated automated techniques that are not only quicker and more accurate, but are also capable of particle analysis at or below the range of resolution of the optical microscope.

During the past 20 years, increased usage of aerosols in industry and medicine and heightened concern over aerosols in air pollution, industrial hygiene, and in manufacturing clean rooms has intensified the search for methods that focus on how a particle behaves when airborne in a field of force rather than how a particle appears under the microscope. Thus, the new definition of particle size known as the aerodynamic equivalent diameter (AED) was established. The AED is formally defined as: The diameter of a hypothetical sphere of unit density having the same terminal settling velocity as the particle in question regardless of its geometric size, shape, and true density (1). The AED is of particular importance for evaluating toxicologic effects because certain particle sizes deposit preferentially in different parts of the respiratory tract.

Lung modeling has shown that particles with aerodynamic diameters (Da) of less than 4 microns are those most likely to be deposited in the tissues of the lungs. Therefore, to accurately determine the fraction of particles that have the greatest potential for lung deposition, i.e. the respirable mass fraction, a method is required that measures the mass of particles having an aerodynamic diameter of four microns or less.

For the purposes of this study, a variety of sampling methods for determining particle size distributions of fine powders were considered. However, most of these techniques were ruled out because they require expensive apparatus, high levels of skill, considerable expenditures of time, or because the principles upon which their operation are based are not fully understood. The method ultimately selected for adaptation to estimate the respirable fraction from the bulk dust was that of liquid sedimentation using the Andreasen sedimentation pipette.

A.H.M. Andreasen is recognized as the individual who pioneered the sedimentation pipette technique in the 1930's for the purpose of resolving particle size distributions of fine materials. To carry out these analyses, Andreasen designed the well known "Andreasen Sedimentation Pipette". The Andreasen sedimentation pipette continues to experience widespread use today due to its simplicity in operation and inexpensive apparatus. The fundamentals of the pipette's

operation are well validated and the method is generally accepted as accurate within $\pm 0.5\%$ - $\pm 3\%$ (3,4,5,6,8,9,11,15).

The criteria for choosing this method are extensive and therefore merit discussion. They are listed as follows:

- 1) The method is applicable to particles in the subsieve size range-i.e. those with a diameter not greater than 50 microns.
- 2) The method yields particle size results cast in terms which are easily converted to aerodynamic diameter.
- 3) The method requires a minimum degree of skill, time, and expense without sacrificing acceptable degrees of accuracy.
- 4) The method is adaptable to a wide range of powders.
- 5) The method gives reproducible results.
- 6) The method provides sufficient results to plot a cumulative size distribution.
- 7) The method provides a distinguishable cutpoint between respirable and nonrespirable particles.
- 8) The sampling procedure does not chemically or physically modify the dust particles so further analyses can be carried out if desired.

PREVIOUS WORK

Many studies have been carried out for the purpose of evaluating the performance of the Andreasen sedimentation pipette. Irani and Callis' report provides an excellent discussion of the more prominent of these investigations, and also confirms the accuracy of the method by comparison with other accepted methods of sampling (9). Irani carried out an intercomparison between the Andreasen pipette method, microscopy, and two sedimentation balances on two samples of hard wheat flour, one fine-grained and the other coarse (28). The microscopic analysis was achieved by electronic counting and sizing. The sedimentation balances were the automatized Gallenkamp balance and the commercial Recording Sedibal. Irani's results showed that the pipette, microscopic, and sedimentation balance data were in close agreement for the coarse flour sample but that the pipette data predicted a finer size distribution than the microscope and sedimentation balances for the fine flour sample. Irani presumed that this deviation was a result of the disturbance generated within the suspension as the samples were withdrawn.

Rabatin and Gale also compared the sedimentation balance and Andreasen pipette with results showing only a slight deviation between the two methods (29).

Wichser and Shellenberger intercompared the Andreasen pipette method with sieve analysis, and air flotation using

the Roller Air Analyzer on a sample of wheat flour (15). They found good agreement between the three methods below the 50 microns, and poor agreement above this size. Significant errors occurred with the pipette analysis when the specific gravity of the medium approached that of the sample. The results of their study demonstrate that the Andreasen pipette is applicable to particles below 50 microns, the sieving method to those above 37 microns, and air flotation to those below 80 microns.

Batel conducted an analysis of the Andreasen sedimentation method and concluded that the pipette is capable of size analysis in the range of 1 to 60 microns but only under conditions of complete particulate dispersion within the medium (30).

Schweyer performed an extensive evaluation and intercomparison of the Andreasen pipette, the hydrometer, the Wagner turbidimeter, and the Roller Air Analyzer methods for particle size analysis (5). Schweyer's results cited the pipette method as the method of choice for determining the particle size distribution of subsieve material by sedimentation techniques. Schweyer also found that the performance of the pipette was dependent on adequate dispersion.

Rendall and Sittert performed a comparison of the Andreasen Pipette Method and the Coulter Counter (Coulter

Electronics), a device which counts and sizes dust particles suspended in an electrolyte solution (19). Their data showed that the two methods predicted similar size distributions for a sample of classified quartz dust, with the Coulter Counter predicting slightly higher proportions of the finer particles. They attributed this trend to the Coulter Counters's definition of particle diameter as being smaller than the Stokes' diameter as defined by the sedimentation analysis.

Although Andreasen's method is well documented in the literature for making quantitative particle size determinations for fine powders, the literature does not indicate that the method has been used for respirable fraction measurements (3,4,5,6,8,9,11,15).

PROJECT OVERVIEW

The purpose of this research was to develop a method for determining the mass fraction of respirable particles in a bulk silica-containing material. The method chosen for evaluation was sedimentation in a liquid medium. To validate the sedimentation results, cyclone respirable measurements were performed on the same dusts, and side-by-side comparisons of the results were made. When available, particle size distribution data provided by the manufacturer was also used as a means of assessing the accuracy of the sedimentation results. On occasion, the manufacturer provided size distribution data but failed to disclose the sampling method. In these cases, comparison between the sedimentation results and the manufacturers' particle size data was carried out although with some dilution of the strength of the comparison.

Thirteen test dusts were analyzed using both the sedimentation and cyclone method. Dusts characterized by various densities, porosity, particle shapes and sizes were selected for analysis so that the range of application for the sedimentation method could be assessed. These dusts included flint, clay, 4 diatomaceous samples, 6 pesticides, and a micronized amorphous silica sample. An additional Arizona road dust sample and talc sample were analyzed using only the sedimentation method and not the cyclone. For the purposes of this report, the diatomaceous samples will be

referred to as diatomaceous compound #1, diatomaceous compound #2, and so forth, as will the pesticides be referred to in the same manner, for example, pesticide compound #1, pesticide compound #2, etc.

The sedimentation method yielded results which predicted the fraction or percentage of particles falling within a specified size range. This percentage of particles was plotted against particle size to predict a particle size distribution for each dust. The sedimentation method measured particle size in terms of Stokes' equivalent diameter. Therefore, determination of the fraction of particles that were in fact respirable required two additional steps 1) conversion of the results from Stokes equivalent diameter to aerodynamic diameter by application of the equation:

$$D_a = D \sqrt{\rho_p} \quad (1)$$

where D_a is the particle aerodynamic diameter (μm)
 D is the particle diameter (μm)
 ρ_p is the particle density (gm/cm^3)

and 2) defining a cutpoint based on aerodynamic diameter which distinguished the respirable fraction of the dust particles from the nonrespirable fraction.

The cyclone method yielded results which defined the dust in terms of two distinct components, the respirable and the nonrespirable fraction. The ratio of the respirable dust sample to the total dust sample represented the respirable

fraction of the bulk material when airborne. The cyclone method directly measured the dust particle sizes in terms of aerodynamic diameter.

Following the analyses of the various dusts, the ability of the sedimentation method to accurately predict the respirable fraction of the bulk test dust was assessed depending on the degree of correlation with the cyclone results and the manufacturers' specifications when available.

Information provided for each test dust includes sedimentation particle size distributions, cyclone respirable fraction measurement data, and physical characterizations such as density, particle shape, and moisture content. Sampling procedures and equipment for both the sedimentation and cyclone analyses are outlined in Appendix A. Appendix C contains photographs of the experimental designs and apparatus for both the sedimentation and cyclone analyses.

METHODOLOGY - SEDIMENTATION METHOD

Sedimentation Apparatus

Cumulative size distributions of the bulk test material were determined by sedimentation techniques. An Andreason Sedimentation Pipette, Fisher Catalog No. 14-232, manufactured by Q Glass Company was the apparatus employed for particle sizing. The Andreasen sedimentation pipette is generally used for determining sub-sieve grain sizes in the 0.5 to 40 or 50 micron range using water as the sedimentation medium. It is frequently relied upon for particle size distribution measurements due to the simplicity in operating the pipette and the relatively low cost of the equipment. The Andreason pipette consists of a 550 milliliter (ml) glass cylinder graduated from 0 to 20 centimeters (cm) and a 10 ml pipette. A polypropylene chuck-type adapter seals the top of the cylinder with the exception of a small opening through which the pipette's lower sampling tube passes vertically. The adapter allows for precise positioning of the lower sampling tube at the desired depth within the glass cylinder. The upper end of the pipette's sampling tube above the adapter is connected to a 10 ml glass bulb by means of a three-way stopcock. Figure 1 shows a diagram of the Andreasen sedimentation pipette.



Figure 1. Andreasen Sedimentation Pipette
(From Andreasen, Reference 32)

Rationale of the Sedimentation Approach

The pipette operates according to theory explained by Stokes' Law. Stokes' Law describes particle motion in a viscous medium. When a particle settles under conditions of laminar flow, Stokes' Law defines its resistance to motion, F_R , as:

$$F_R = 3 \pi \mu v d \quad (2)$$

where μ is the viscosity of the medium (gm/cm sec)
 v is the settling velocity (cm/sec)
 d is the particle diameter (cm)

Letting g be the acceleration due to gravity (cm/sec²)
 and m the mass of a particle (gm), then

$$F = m g \quad (3)$$

For spherical particles of mass m ,

$$m = \frac{\pi (\rho_p - \rho_m)}{6 d^3} \quad (4)$$

where ρ_p is particle density (gm/cm³)
 ρ_m is fluid density (gm/cm³)

Equating the gravitational force with the resisting force and solving for the settling velocity (v), the equation becomes

$$v = \frac{d^2 (\rho_p - \rho_m) g}{18 \mu} \quad (5)$$

Equation (5) represents the settling velocity of a particle of diameter (d) and density (ρ_p) which is settling in a medium of density (ρ_m).

The relationship between particle diameter and the distance a particle falls by gravity is determined by the equation:

$$d = \left[\frac{(18 \mu H)}{(\rho_p - \rho_m) g t} \right]^{1/3} \quad (6)$$

where t is the time the particle falls (sec)

H is the distance the particle has fallen in time t (cm)

i.e. the distance between the fluid surface and the pipette tip

If the pipette flask initially contains a liquid medium with a uniform distribution of particles, then all particles having a diameter equal or greater than d will settle out of the suspension at height H after time t . For example, for d , H , and T values of 50 microns, 20 centimeters, and 80 seconds respectively, all particles greater than 50 microns in diameter will have settled out of

the suspension at a height of 20 centimeters after 80 seconds. Samples drawn out at height H in successive intervals of time will show smaller and smaller numbers of larger particles as the larger particles settle out at a faster rate than the smaller particles. Figure 2 illustrates the sedimentation procedure at subsequent time intervals.

This relationship can be used to determine the cumulative size distribution by mass of certain materials. Samples can be collected at various times t at height H using the sedimentation pipette. Each sample is evaporated to dryness and the particle mass of each sample is determined. The weight of each sample is expressed as a percentage of the first sample drawn to give the percentage of the test dust having particle sizes smaller than the largest particle contained within that sample. A cumulative size distribution by mass is then obtained by plotting the cumulative percentage of particles by mass having diameters less than a given particle size against the Stokes' equivalent diameter provided by equation (6).

Assumptions of Stokes' Law

To properly use Equation (6) in predicting particle size distributions by the sedimentation method, it is essential to consider the applications and limitations of the theory. The solution of Stokes' Law assumes a viscous,

SCHEMATIC OF SEDIMENTATION TEST

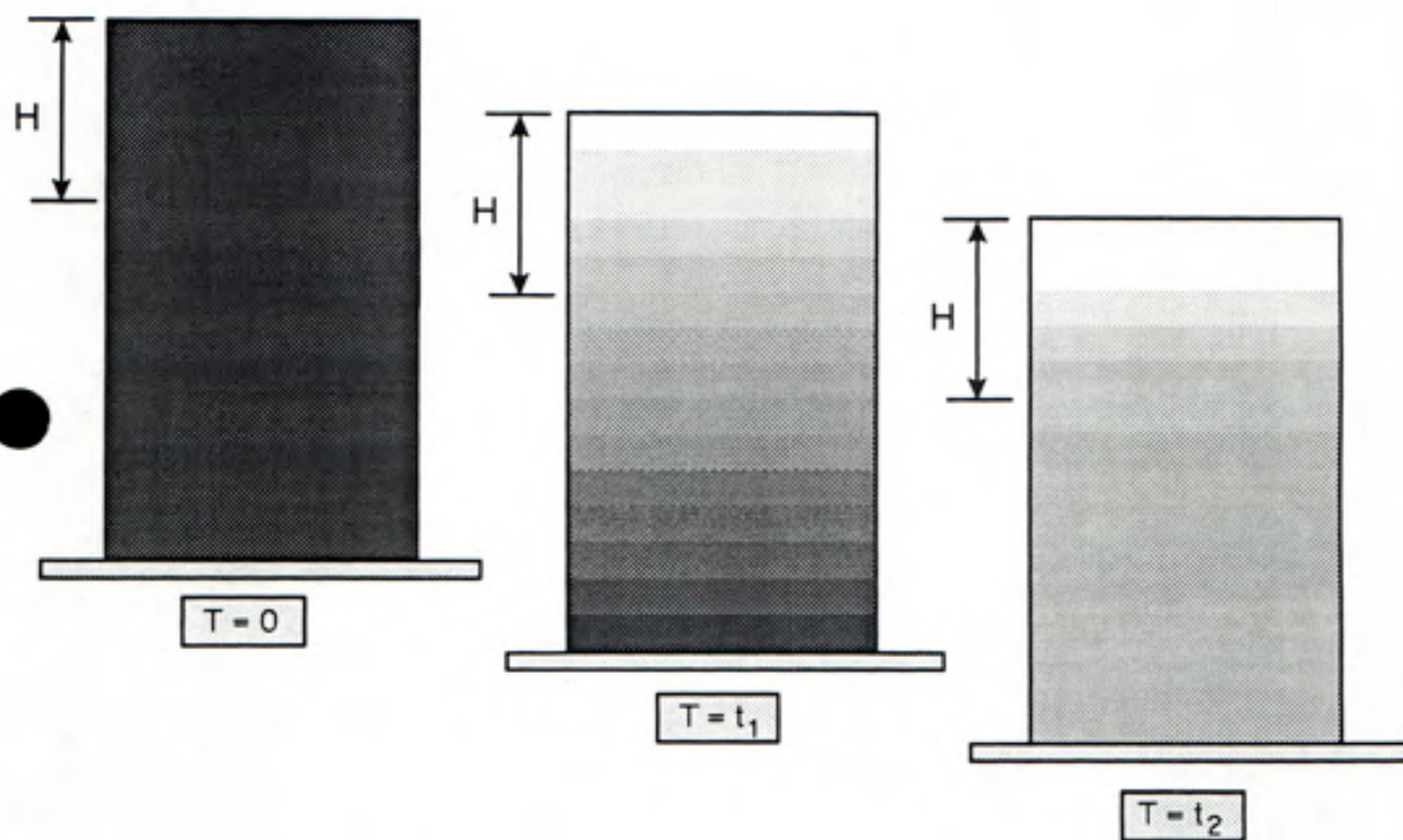


Figure 2. Schematic of sedimentation test.

continuous, and infinite medium with streamline motion of spherical particles. A discussion of each assumption follows. Davies' classic article on the subject is highly recommended (21). More complete derivations of Stokes' Law can be found in the literature (14,16,17).

A. Viscous Medium

The application of Stokes' Law assumes that the resistance to the particles' motion is due to the viscosity of the fluid and that the inertia of the fluid is negligible (3). This assumption holds true when the particles are sufficiently small. In cases of increasing particle size however, inertial forces take on greater significance as a wake starts to develop behind the particle.

The ratio of inertial forces to viscous forces is defined by the Reynolds number. Davies reports that for spherical particles settling through water, Reynolds numbers of 0.074, 0.38 and 0.82 correspond to 1%, 5%, and 10% errors respectively in the application of Stokes' Law (21). Davies further estimates that for particles having densities of 2.5 gms/cm³ settling in water, the particle diameters associated with Reynolds numbers of 0.074, 0.38 and 0.82 are 45, 79 and 104 microns, respectively. These data are summarized in Table I. Thus, for the purposes of this study, Davies' data show that the sedimentation method is best suited to the

sampling of particles having diameters less than 50 microns when using water for the sampling medium.

TABLE I.

Error Associated with the Application of Stokes' Law for Spherical Particles Settling through Water at 20°C *

Reynolds Number	Particle Size (microns)	Error
0.074	45	1%
0.380	79	5%
0.820	104	10%

* Assumes particle density of 2.5 gm/cm³.

B. Continuous Medium

Stokes' Law assumes that the fluid is a continuous medium. When the diameter of the falling particles approaches that of the fluid molecules, a condition of slippage occurs, i.e. the particles begin to slip between the molecules of the fluid. This phenomenon is observed when particles have diameters less than one micron. The Cunningham correction factor (C_C) has been adopted to correct for this effect. In the sedimentation method proposed by this study, slippage is insignificant and the correction factor is therefore unwarranted.

C. Infinite Medium

With viscous flow the interference caused by a particle continues large distances into the medium. If other particles are settling nearby, then the resistance of the medium to these particles is lowered because of the movement induced within the medium by the particle in motion. For this reason, a particle will settle at a higher velocity when it travels with a group of particles rather than when it settles individually. Also, when two particles of equal sizes settle along the same axis, the trailing particle will settle at a higher velocity than the leading particle and the two will eventually collide. If the particles have unequal diameters, both particles will settle with an increase in velocity due to the aerodynamic interaction between the them. When the trailing particle is larger than the leading one, its increase in velocity is less than that for the leading particle (14).

Much effort had been dedicated to the study of the influence of the concentration of a suspension upon the sedimentation velocity for spherical particles in the viscous flow region. The issue of determining a suitable concentration is somewhat obscured by the fact that the net effect is actually the result of two opposing forces. A particle settling within the sedimentation flask will experience a downward drag force owing to the flow field of neighboring particles. However, given that the fluid is

contained in a confined vessel, an upward flow also occurs to compensate for the downward particle motion. The resultant effect is of a smaller order of magnitude than either phenomenon, because the two opposing forces tend to balance out. In general, the downward velocity of the particles in an enclosed vessel is less than they would be in an infinite medium (21).

When using the sedimentation method in a liquid medium, the validity of Stokes' Law is assured only when the particulate concentration does not exceed 1-2% by volume. Particle interactions previously described will occur with concentrations in excess of this range with a resultant biasing of the sampling data. The literature shows conflicting opinions concerning the exact concentration where Stokes' Law ceases to apply.

D. Streamline Motion

Stokes' equation applies only when the motion of the particle in the fluid is streamline. A correction should be made at higher velocities. Rose demonstrated the validity of Stokes' Law to within 1% when the Reynolds number is no greater than 0.1 (8). His calculations show that for a solution of water at 25°C and silica particles having densities of 2.5 gm/cm^3 , the upper limiting diameter for streamline motion is 50 microns. The 50 micron designation does not represent a sharp cutpoint but rather a diameter

where errors in the application of Stokes' Law begin to arise. The magnitude of the error increases with particle size, for example, a 15% error is predicted for sedimentation analyses of 120 micron particles.

The lower limiting diameter for streamline motion is 1 to 2 microns. Below this limiting diameter particles begin to feel the effect of the random molecular movement of the suspending fluid and consequently experience Brownian motion, thus Stokes' Law becomes inoperative. Particles having diameters less than 1 micron are also particularly vulnerable to the effects of even slight convection currents.

E. Spherical Particles

The final assumption in the application of Stokes' Law is that the particles are perfect spheres. However, the sedimentation method can usefully be applied to particles of most any shape as long as it is recognized that the diameters predicted are equivalent diameters.

Studies have demonstrated that the relation holds quite well for particles which vary appreciably from sphericity. Andreasen showed that in the viscous flow region, irregularly shaped particles with compact shapes tend to settle at the same velocity as spheres of equal density and volume (21). This behavior extended to particles possessing sharp edges as well.

Adapting the Method to the Study

Certain features of the Andreasen sedimentation pipette technique can be modified depending on the user's sampling objectives and the material under investigation. The most significant of these features are 1) the choice of the sampling fluid, 2) the procedures employed to ensure complete dispersion of the solids within the sampling suspension, and 3) the sampling height necessary for collecting the particles of interest. Each of these issues is highlighted in the following discussion on adapting the Andreasen pipette method to the call for respirable fraction measurements.

A. Selection of the Sedimentation Fluid

The success of any particle size measurement technique is dictated by its ability to achieve the proper dispersion of the particles into their working units. For the Andreasen sedimentation pipette, this meant choosing the proper sedimentation fluid and faithfully incorporating sound dispersion practices.

A solution of distilled and deionized water was selected as the sedimentation fluid for the experimental analyses. Distillation assured that the liquid was free from foreign materials which could influence test results, and

the deionization process eliminated electrostatic charges within the medium which could influence particle behavior. The silica dusts were not soluble in the water, nor did they chemically react with it. The relationship of the viscosity and density of the water ($0.01 \text{ dyn. second/cm}^2$ and 1.0 gm/cm^3) with the density of the test dust ($2.0\text{--}2.7 \text{ gm/cm}^3$) was such that the particles attained an acceptable settling velocity during sedimentation. This relationship also allowed for a majority of the settling to occur within the Stokes' region, i.e. within the viscous flow regime and outside the region dominated by Brownian motion (9). Other benefits of the water medium were low cost, nonflammability, nontoxicity, and ready availability.

B. Dispersion Procedures

Dispersion of the suspension was the single most important parameter in ensuring the integrity of the sedimentation results (9,12). Dispersion was contingent upon the choice of proper sampling fluid as previously discussed, the addition, when necessary, of the proper dispersant in the correct quantities, the proper particle concentration within the suspension, and the temperature of the suspension.

The degree of dispersion was determined by microscopic examination of a slide prepared from a drop of the dilute suspension. Well-dispersed suspensions were characterized by

evenly spaced particles that moved freely within the solution without adhering to or clustering with other particles. Poorly dispersed suspensions showed particle agglomeration where individual particles failed to move independently of particles.

The distilled and deionized water did not always completely disperse the test dust. In these situations, a small amount of dispersing agent was added to the suspension. This procedure had the effect of lowering the surface tension of the liquid, and therefore improving the wettability of the surface of the particles. The choice of the dispersing agent was simply a matter of trial and error, and usually several dispersing agents were tested before observing the desired level of dispersion. Dispersion agents used for this experiment included soap, ethyl, and isopropyl alcohol. Only a fraction of a per cent of a dispersing agent was added so corrections for viscosity and density of the water were not warranted.

Another important factor in assuring good dispersion was the concentration of the dust within the fluid. The literature recommends a concentration ranging from 0.25% to 2.0% by volume, however there is little agreement as to which of these concentrations is best. Andreasen recommends 2% by volume, Irani and Callis agree on 0.2-0.5% by volume, and the pipette manufacturer recommends 1-2% by volume (8,9,22). The difficulty in obtaining the correct

concentration is rooted in the need to meet the dual requirements of having a concentration low enough to allow unhindered settling of the particles but one high enough to guarantee detectable quantities, particularly for the finer particle sizes. For the purposes of this study, concentrations of 0.25% to 1.25% by volume were used with no signs of agglomeration or interference of particle settling. The author does not recommend concentrations at the lower end of this range for reasons previously discussed.

As dispersion behaviors will vary from one dust to another, it is good practice to perform the sedimentation analysis at several concentrations and with more than one dispersing agent. If all analyses reflect similar results, it is likely that dispersion is complete; likewise if conflicting results are obtained then the analysis with the highest percentage of fines should indicate the technique with the most complete dispersion.

Some dusts, talc for example, showed partial dispersion within the water medium. For these dusts, one particle component readily dispersed within the medium and the other component remained on the fluid surface in spite of extreme agitation of the mixture. In these cases, dispersion was achieved by first preparing a slurry of the water and dust, and then adding the slurry to the water within the pipette. A small amount of dust was added to a few drops of water, and the mixture was stirred until a thick paste was formed.

This process was repeated until all of the sample was incorporated into the paste. At this point, enough water was added to the slurry to allow for transfer to the sedimentation flask.

A final precaution taken to ensure for proper dispersion was to allow the water to reach thermal equilibrium with the test environment before sampling commenced. This was achieved by storing the water in the laboratory for several days prior to sampling. This process minimized the introduction of thermal currents by temperature variations which would prevent the free settling of the individual particles. This procedure is a relatively simple and inexpensive alternate method for temperature control in comparison to the use of thermal insulators that are often called for in guidelines for carrying out sedimentation analyses with the Andreasen pipette (8).

C. Increasing the Sensitivity of the Method

A major disadvantage associated with the Andreasen sedimentation pipette is the significant amount of time required for the sizing of fine particles when following the standard procedure of using a water medium and the designated 20 cm sampling height. This was of particular concern for this project since determining the respirable fraction called for sizing particles having aerodynamic diameters down to 3 or 4 microns. For a silica dust, Stokes'

Law predicts that these particles would require a sampling time of at least 10 hours.

Observation of Stokes' Equation as defined by equation (6) suggests that several variables can be feasibly altered with the net effect of reducing the sampling time (t). These are the sampling height (H) and the viscosity of the medium (μ) which in turn would change the medium density (ρ_m). Variations on each of the above were investigated. The sampling strategies employed included 1) reducing the sampling height (h) from 20 cm to 5 cm. Data from a standard sampling run for the same dust (i.e. using a water medium and an initial 20 cm sampling height) was used as a standard of reference, and 2) using hexane in place of water as the sampling medium. This substitution would show a decrease in viscosity and density relative to water and would allow for more rapid settling of dust particles.

Sampling at a height of 5 cm was carried out in a water medium using the Andreasen method. As predicted by Stokes' Law, this procedural modification greatly reduced the 10 hour sampling time for 3.5 micron particles (D_a) down to 45 minutes. Table II, which reflects sampling time and particle diameter for water-based runs, shows that the diameter of the largest particle present in each sample decreases at least by a factor of two when reducing the initial sampling height from 20 to 5 cm. This effect becomes more pronounced with time.

TABLE II.
Comparison of Sampling Times
at 5 and 20 Centimeter Sampling Height

Time (seconds)	<u>Particle Diameter Sampled (microns)</u>	
	20 Cm	5 Cm
20	121	51
120	45	17
700	18	6
1200	14	4
1800	11	3
2700	9	2
3650	7	2

Figure 3 shows that the accuracy of the data also seems to improve with the shorter sampling height, perhaps because now there are at least two points with mass data less than 50% of the total mass. Agreement is better with the 5 cm height both in median diameter and geometric standard deviation.

Conducting the experiment with hexane as the medium yielded unexpected results. Upon contact with the hexane fluid, the silica particles immediately agglomerated into clusters and settled to the cylinder bottom, thus eliminating the possibility of particle sampling. A possible explanation for this occurrence is the effect of electrostatic forces. Because the sample readily dispersed in water, a polar solvent, it can be assumed that the sample is also polar. Thus electrostatic forces between the water molecules and the sample molecules helped to overcome the

COMPARISON OF SAMPLING HEIGHT

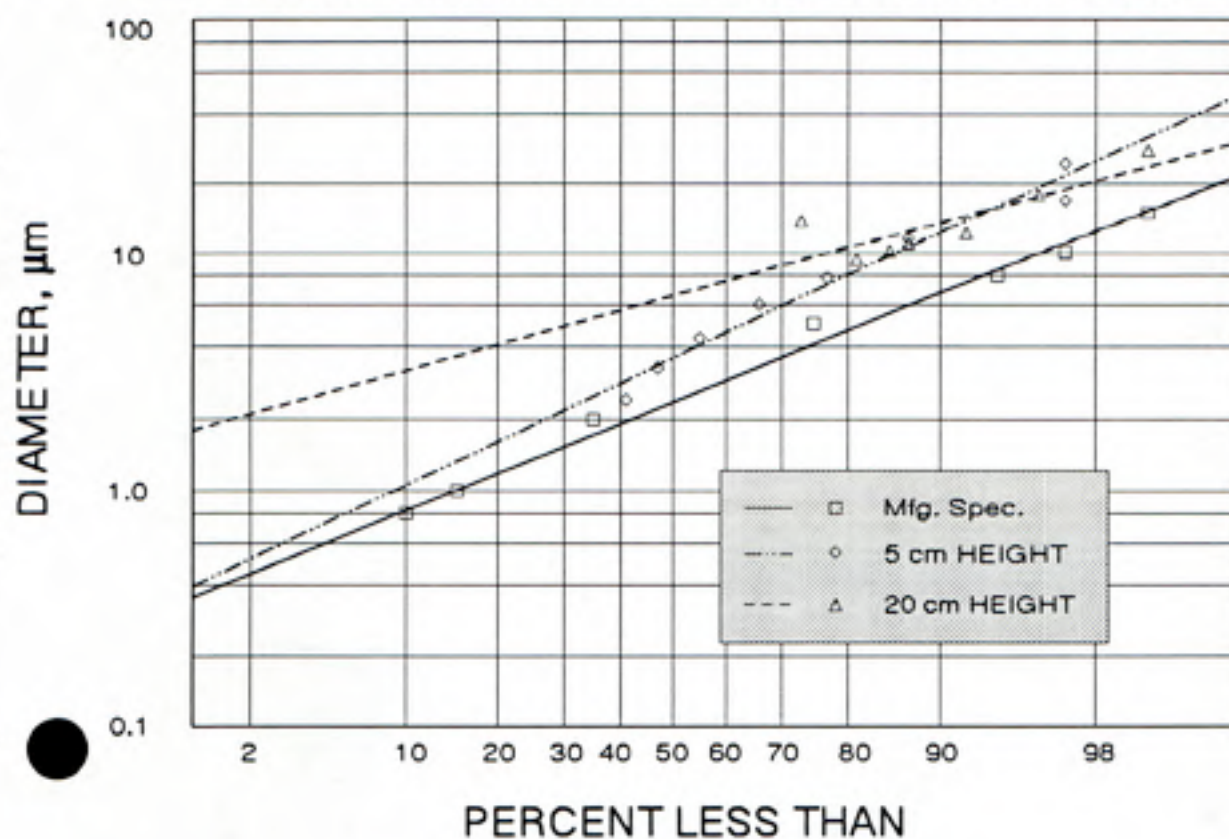


Figure 3. Comparison of sedimentation data at a sampling height of 5 and 20 cm for Micronized Amorphous Silica.

bonds between sample particles, consequently preventing particle agglomeration.

In contrast, the hexane molecules, which are nonpolar, failed to interact with the sample particles, therefore allowing the sample particles to bond together to form large clusters with settling times of only a few seconds. This view was supported by mixing the sample with several mediums of a polar and nonpolar nature. Even so, calculations using polar alcohols such as methyl, ethyl, and isopropyl alcohol for the medium indicated that the best approach to shorter sampling times was to reduce the initial sampling height to 5 cm rather than substituting these substances for water.

Based on the results of this investigation, the 5 cm initial sampling height with the water medium was incorporated into the experimental design for the sampling of all test dusts.

Experimental Procedure for Sedimentation Method

After completing the dispersion analyses, the sampling method involved preparation of a suspension by mixing a known quantity of test material and distilled, deionized water within the glass cylinder. The amount of test material added ranged from a concentration of 0.25% to 1.25% by volume.

The mixture was then agitated for two minutes and samples of the suspension were then pipetted during

sedimentation by applying suction to the upper end of the pipette with a rubber bulb. The samples were drawn into the 10 ml glass bulb at successive time intervals, with the actual sampling interval running approximately 20 seconds (4). Turning the stopcock a quarter revolution and applying pressure to the rubber bulb allowed the contents of the 10 ml glass bulb to drain via a faucet-shaped mechanism into a pre-weighed aluminum evaporation dish. The aliquots were then evaporated to dryness in a drying oven. The samples were removed from the oven, allowed to cool and then weighed on a Mettler Model HL 52 balance. Time measurements were conducted with a Heurr stopwatch. All sedimentation runs were carried out at 22°C.

Table III shows a typical sampling run for the sedimentation analysis.

TABLE III.
Typical Sampling Run for Sedimentation Analysis

Sample	Dry Weight (gm)	Time (sec)	Height (cm)	Diameter (micron)
1	0.236	10	5.0	74.60
2	0.166	120	4.6	20.66
3	0.125	300	4.2	12.48
4	0.087	720	3.8	7.66
5	0.067	1200	3.4	5.62
6	0.057	1500	3.0	4.72
7	0.047	1800	2.6	4.01
8	0.042	2100	2.2	3.41
9	0.035	2400	1.8	2.89
10	0.028	2700	1.4	2.40
11	0.019	3600	1.0	1.76

Development of Cumulative Size Distributions

The weight of the first sample represented the initial weight of the test material. The percentage by weight of specific groups of grain sizes were calculated by dividing the weight of each sample into the initial weight of the test material, i.e. the weight of the first sample. The Stokes equivalent diameter of the fallen particles was then determined by application of Stokes' Law as described by equation (6). Since it took about 20 seconds to siphon the sample, the midpoint of the interval was used for calculating particle size.

Each sample drawn has a smaller particle size than that corresponding to the equivalent diameter predicted by Stokes' law because all particles of larger size will have fallen below the level of the tip of the pipette's sampling tube.

Cumulative size distributions by mass were then obtained by plotting the particle mass of each sample against the diameter provided by the equation on log-probability paper. Each dust curve required a certain degree of extrapolation at the lower portion of the curve depending on the number of data points available for the smaller particles. Longer sampling times, such as 90 minutes, and/or higher particle densities, such as 2.65 gms/cm³ provided more data points at the lower portion of

the curve and thus required less extrapolation. In contrast, dust with low densities, such as 2.0 gms/cm^3 , and/or shorter sampling times, such as 60 minutes, provided fewer data points at the lower end of the curve and thereby required greater extrapolation.

Defining the Respirable Cutpoint

Determining the respirable fraction of the test dust using the sedimentation data first required developing a exact cutpoint which distinguished between respirable and nonrespirable particles. The American Conference of Governmental Industrial Hygienists (ACGIH) defines respirable particulate mass as follows:

"Respirable Particulate Mass consists of those particles that penetrate a separator whose size collection efficiency is described by a cumulative lognormal function with a median aerodynamic diameter of 3.5 microns ± 0.3 microns and with a geometric standard deviation of 1.5 ± 0.1 ." (24)

Based on the ACGIH guidelines, an aerodynamic diameter of 3.5 microns was selected as the respirable cutpoint for the sedimentation data (31). Application of the equation ($D_a = D\sqrt{\rho_p}$) provided conversion of aerodynamic to actual diameter depending on the dust particle density. The resulting value was then applied to the sedimentation cumulative size distributions and the percentage of particles with diameters less than the chosen value was taken to be the respirable fraction of the bulk sample.

Evaluation of the Role of Bias

Although the Andreasen sedimentation pipette is generally accepted as providing an accuracy of $\pm 0.5\%$ to $\pm 3\%$, it is important to address potential sources of bias and the direction of their likely effect on the observed results. When the sources of error are recognized and controlled for by periodic checking with other methods, sedimentation is considered by many to be the best method for particle size analysis.

A consistent criticism of the Andreasen sedimentation pipette is that the withdrawal of the sample removes a portion of the suspension with a thickness in excess of one centimeter, and subsequently disrupts the free settling of the particles that are to be sampled next. Various individuals have modified the design of the Andreasen pipette with the intent of reducing the effects of these disturbances, although the author is not familiar with these apparatus (8,10,23). It has also been noted that the pipette may actually collect particles a significant distance below the pipette tip, particularly when the sample is drawn too rapidly and when extreme pressure is applied to the bulb. This tendency is more pronounced with the very fine materials and will serve to overestimate the results for the per cent remaining in suspension. This is not as likely to occur for the coarser grained particles. Drawing the sample at a slow and steady rate over a 20 second

interval is the best approach to minimizing these effects (4).

Another source of error arises when the sedimentation method is carried out without the proper degree of dispersion. This effect could bias the size frequency curve in either direction, depending on the dispersion behavior patterns. If poor dispersion is due to the clumping of particles, then the bias would be towards the larger particles at the expense of the smaller ones since individual particles behave with densities and diameters much higher than their true values. This result would be an underestimation of the percentage of respirable particles. On the other hand, if the poor dispersion is due to excessive concentration, then the percentage of fines would be overestimated because of the hindered settling effects (13).

Although inadequate dispersion is the most frequently occurring source of error when employing sedimentation sampling methods, it should not discourage individuals from employing this method. Poor dispersion rarely occurs with subtlety and thus is easy to recognize and control for. In most cases, improper dispersion results from an excessive concentration of particles within the suspension and/or the presence of electrostatic charges. In the former case, decreasing the particle concentration, and in the latter, addition of a dispersant is all that is required to achieve

proper dispersion. Analysis of a few drops of the suspension under the microscope should confirm whether the proper degree of dispersion has been obtained. However, in the event that sampling is carried out under conditions of poor dispersion, oftentimes the error will clearly manifest itself in the sampling data and the size frequency curve. Table IV show data collected for a talc sample under conditions of poor dispersion. Evaluation of the data show significant increases rather than decreases in mass from one sample to the next. Significant increases from one sample to the next are clear evidence for the occurrence of poor dispersion. Hinkley provides an excellent discussion on additional indicators of poor dispersion (12).

TABLE IV.
Sedimentation Data Collected
Under Conditions of Poor Dispersion

Sample #	Sample Collected Dry Weight (Gms)	Sampling Time (Secs)	Sampling Height (Cms)
1	0.047	10	5.0
2	0.054	120	4.6
3	0.047	300	4.2
4	0.050	720	3.8
5	0.038	1200	3.4
6	0.048	1800	3.0
7	0.041	2400	2.6
8	0.041	3390	2.2

A final bias associated with any sedimentation pipette is that between successive samples, a small amount of the

suspension remains in the pipette stem between the operating tap of the pipette and the pipette's tip. This space between the pipette tap and the tip is known as the dead space, and the error it introduces is called the dead-space error (13). It is generally agreed that the dead-space error is probably too small to be of any consequence(4). In the case of the Andreasen sedimentation pipette, the pipette stem's channel is so narrow that significant dead-space errors are unlikely.

METHODOLOGY - RESPIRABLE FRACTION MEASUREMENTS

Elutriation Using a Cyclone

Sedimentation respirable results were validated by side-by-side comparisons with cyclone respirable measurements performed on the same dusts.

The 10-mm nylon cyclone precollector followed by a preweighed filter is the most widely used method for determining respirable mass in the workplace. The cyclone's operation is based on a recognition of the size-selecting characteristics of the human respiratory tract (18). A sampling pump is used to draw air through the cyclone via an inlet that is tangential to the cyclone's cylindrical section. The geometry of the inlet forces the air to spin around the cyclone several times before exiting the top and then passing through the filter. The respirable particles are carried with the air stream and deposited on the filter, whereas the larger particles are propelled out of the airstream by centrifugal force and either deposited on the cyclone walls or dropped into the removal section or grit pot at the cyclone's bottom (25). Lippman's article offers a unique and up-to-date perspective of size-selective sampling and the cyclone, as well as providing scientists of a more zealous nature with 183 further references (26).

Respirable mass sampling is routinely carried out in industrial settings to determine compliance status with

federal respirable mass standards. At this time, the U.S. enforces respirable dust standards for occupational exposure to silica, coal, talc, a few other mineral dusts, and nuisance particulates. Respirable dust standards for silica are shown in Appendix B.

Respirable dust concentrations within a workplace can be determined by sampling a volume of air with the cyclone and filter assembly and then dividing the net weight of dust collected on a filter by the total volume of air sampled. (Flow rate X sampling time = total volume of air sampled.) Respirable mass sampling results are only valid for dusts that are absorbed in the alveolar region of the lung. The ACGIH provides guidelines for Particle Size-Selective TLVs for respirable crystalline silica (18, 31). The Particle Size-Selective TLV for materials which are hazardous when deposited in the gas-exchange region of the lungs is expressed as a Respirable Particulate Mass TLV. The ACGIH defines the respirable particulate mass in quantitative terms as follows:

"Respirable Particulate Mass consists of those particles that penetrate a separator whose size collection efficiency is described by a cumulative lognormal function with a median aerodynamic diameter of 3.5 microns ± 0.3 microns and with a geometric standard deviation of 1.5 ± 0.1 ." (24)

This definition is intended to provide an acceptable level of performance for the respirable sampling with the cyclone.

For the research undertaken here, respirable mass sampling was used to determine the respirable fraction of an airborne dust. These results were obtained by aspirating a known amount of dust within a chamber, and taking side-by-side samples of respirable dust and total dust, i.e. dust collected on a filter without a cyclone precollector. The ratio of respirable dust to total dust represented the respirable fraction, as defined by the ACGIH.

Experimental Cyclone Design

Respirable mass samples were collected on 37 mm polyvinyl chloride filters (PVC) having a nominal pore size of 5 microns. The rationale for selecting this filter type was due to anticipation that the samples might be analyzed in the future for free silica. If free silica analysis is not expected, use of standard 0.8 micron membrane filters is recommended.

Sampling was conducted within an acrylic chamber having a volume of approximately one cubic meter or 37 cubic feet. The chamber was sealed from potential leaks to ensure integrity of the system. The sampling train consisted of one open face sampler and three two-stage "respirable" dust samplers at a vertical orientation 40 centimeters from the chamber bottom. The four samplers were placed in a circular fashion in the center of the chamber with the open-face filter and the inlets of the 3 cyclones facing toward the

circle's center. Each sampler was individually connected by flexible tubing to a Doerr vacuum pump, located outside the chamber. The system was designed to provide a flow rate of 1.7 liters per minute (lpm), the flow rate recommended by the ACGIH for respirable mass sampling. Dwyer RMB 50 rotameters were placed in line to measure pump flow rates. The rotameters were calibrated against a primary flow standard. Figure 4 shows the experimental design for the cyclone respirable fraction analyses.

The open face sampler consisted of a PVC filter loaded into a standard Lucite filter holder. This sample represented the "total" airborne dust concentration. The "respirable" dust samplers consisted of 10-mm nylon cyclones and 37-mm cassettes loaded with the 37 mm PVC filters. These samples represented the "mass respirable concentration" of the total airborne material. All filters, respirable and open-face were supported by cellulose back up pads to prevent breakthrough. The cassettes were sealed from leakage with strips of tape.

Experimental Procedure for Cyclone Sampling

Each run was initiated by coating the inside of the chamber with a known amount of the test material. Failure to precoat the chamber resulted in inconsistent sampling data which predicted unreasonably low respirable and total dust levels when considering the actual amount of dust present.

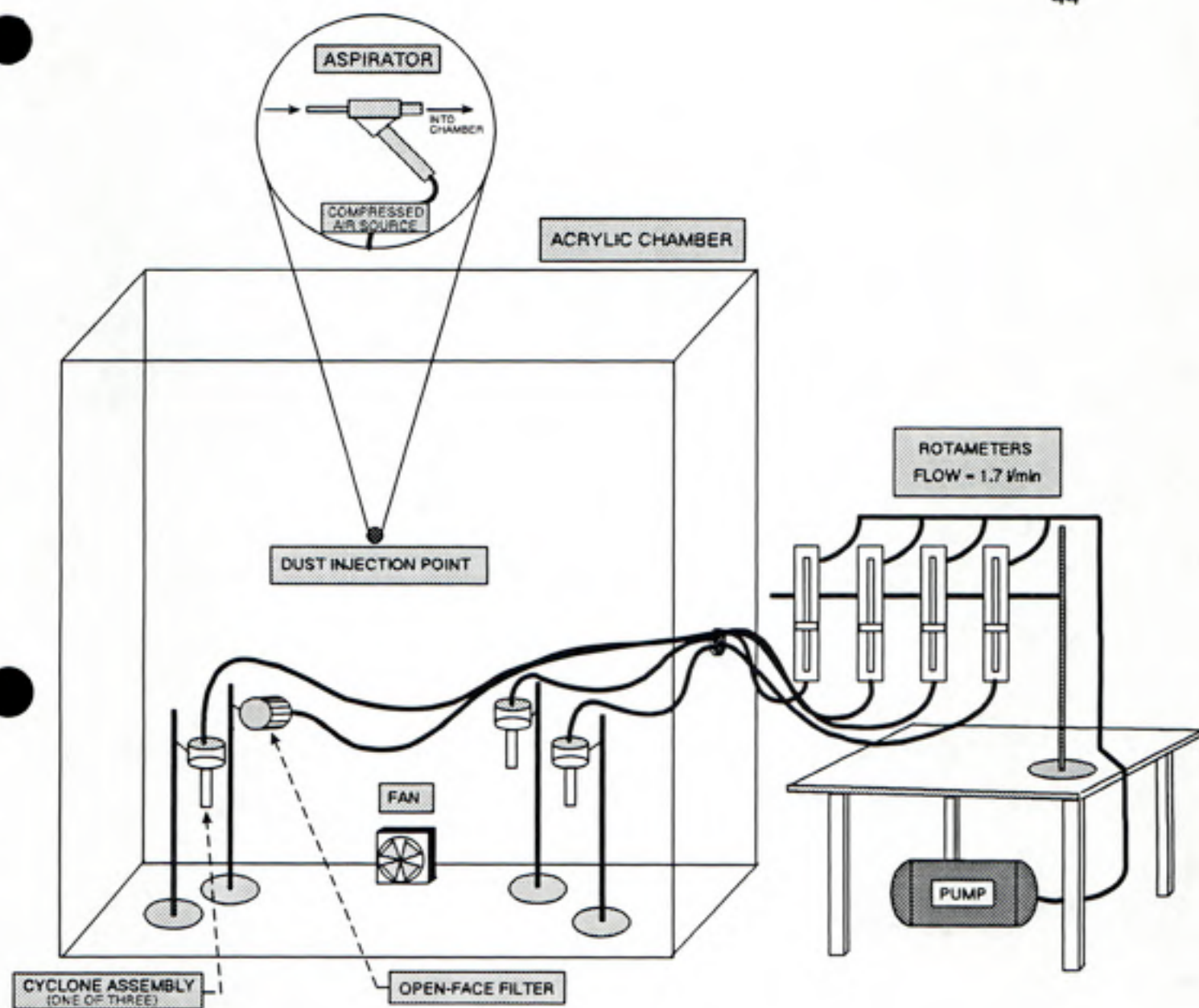


Figure 4. Cyclone Experimental Design.

Upon injection into the chamber, the tendency seemed to be for the particles to move towards the walls and to occupy all free surfaces before developing a uniform dust cloud within the chamber. This observation was further supported by data collected following an initial chamber precoating, which yielded consistent amounts of dust loading on the filters and total dust values in closer agreement with theoretical expectations.

Following the initial precoating, a weighed amount of the bulk test material was dispersed in the chamber. Dispersion was accomplished either by aspirating the dust through a venturi injector into the chamber or in the case of dusts having an appreciable amount of very large particles, by air flotation of the material from a beaker placed on the chamber bottom immediately in front of a mixing fan. After dispersion, a mixing fan was allowed to operate for an additional minute to ensure a uniform distribution of the dust cloud within the chamber. The fan was then turned off and sampling commenced at a flow rate of 1.7 lpm. Actual sampling time varied from 5 to 21 minutes depending on the amount of dust injected into the chamber. Sampling was discontinued if excessive dust loading appeared to be occurring on the open face filter. A Heurr stopwatch was used for all time measurements. Sampling was carried out at room temperature, approximately 22 ° C.

The net weight of dust collected on each filter was determined by weighing the filters before and after each test run on a Mettler Model HL 52 balance accurate to 10 micrograms. To adjust for fluctuations in the final digit during weighing, the result was recorded as the average of five successive readings. Variations greater than 30 micrograms were not accepted; however, variability was usually much lower. The accuracy of this method was confirmed by weighing a 100 microgram standard weight prior to each weighing session.

The high static electrical charge carried by the membrane filters greatly interfered with the weighing process. This problem was overcome by passing the filters over a Po-210 static eliminator (Nuclear Products Co. Model 2U500) prior to each weighing.

The ratio of each of the three respirable dust samples to the total dust sample was calculated and the respirable fraction of the bulk material when airborne was taken to be the average of the three ratios.

Following weighing, the filters were resealed and held for possible future free silica analysis. This analysis would be carried out in accordance with NIOSH Analytical Method #7500 and the sampling was conducted to be in conformance with this method (27).

RESULTS

Sedimentation Data - Cumulative Size Distributions

Sedimentation analyses were carried out on a total of 15 different dusts. One run was conducted for the pesticide dusts, diatomaceous compound #4, and the Arizona road dust, while a minimum of two runs were performed for the remaining dusts. Figures 5-25 show the cumulative size distributions of the bulk material with the percentage by weight equal to or less than a given particle size plotted as a function of particle size (microns). A regression analysis was performed on the data and is plotted as the regression curve shown on the figures. Also shown on the figures are general characteristics of the dusts, as well as the geometric mean and the geometric standard deviation (GSD) of the particle sizes. The geometric mean represents the value with the greatest frequency of particles, and the geometric standard deviation quantifies the particle size variability or relation to the mean. For example, a GSD of 1.0 indicates no variability in particle size, while a GSD of 2.0 represents relatively high variability.

Most samples appear to approximate a log-normal distribution. For dusts that significantly diverged from a log-normal distribution, it was speculated that particle agglomeration may have been occurring. This was also assumed to be the case for dusts that showed an increase in

sample mass from one sample to the next, or had suspiciously high values for the geometric mean particle diameter. For dusts which exhibited these tendencies, the sedimentation analyses were reconducted but with the addition of a known dispersing agent. In all cases, the follow-up experiments yielded data that 1) showed a subsequent decrease in sample mass from one aliquot to the next, 2) predicted a higher respirable fraction than the initial sedimentation run, and 3) that more closely approximated a log-normal curve. These observations were taken as indications that complete dispersion had not been achieved in the original test run.

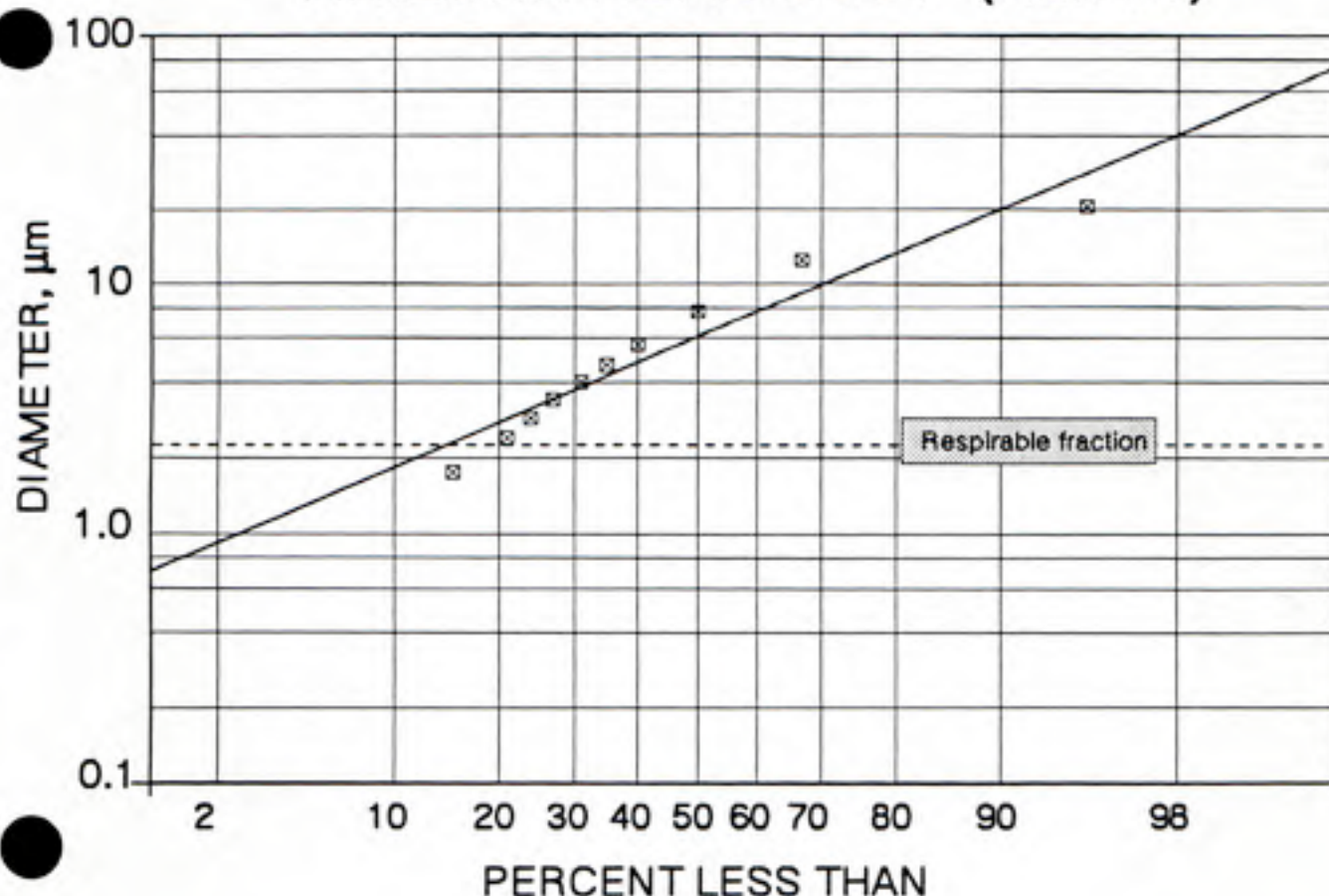
The sedimentation method shows high levels of reproducibility $\pm 1\%$ in predicting the respirable fraction of the bulk material when carried out with adequate dispersion. Varying the suspension concentration by volume within the range of 0.5% to 1.2% did not affect the respirable prediction outside the 1% range of error. It is of interest to note that the sedimentation technique continues to show high reproducibility even when the conditions necessary for carrying out the analysis are not met. For example, consecutive runs carried out on the clay sample in a flocculated state predicted the same respirable fraction value of 10% $\pm 1\%$. These data indicate a state of consistency with the pipette method when repeat analyses are performed on the same dust under the same conditions.

Reproducibility in calculated values of the geometric mean particle diameter and the geometric standard deviation was also achieved for runs with good dispersion. The maximum variance for the geometric mean was 0.5 micron, while that for the geometric standard deviation was 0.38.

Actual concentrations for the first aliquot pipetted for each run were compared to theoretical concentrations of the original dust/fluid suspension. The ability of the first sample to predict the actual initial concentration is of special importance since it is the basis for all subsequent sample calculations. For the 6 pesticide compounds and the Arizona road dust, the actual concentration of the first sample ranged from 48-86% of the theoretical concentration. For the remaining dusts, correlation was much closer with actual/theoretical concentration ratios of 90% and above for 7 of the 8 dusts.

Diatomaceous compounds #1 and #3 showed an increase in sample mass from the first aliquot drawn to the second aliquot drawn. This tendency was observed for at least one of the sedimentation runs for each dust, even when adequate dispersion had been achieved. A possible explanation for this occurrence is that for these particular dusts, an acceptable amount of time was not allowed to elapse before collecting the second aliquot. Several authors recommend sampling time scales with time (t) values that give a $\sqrt{2}$

ARIZONA ROAD DUST (Run #1)

Physical Characteristics

Density - 2.65 gms/cm³
 Particle shape - spheres and irregular

Sedimentation Conditions

Medium - water
 Sampling height - 5 cm
 Sampling time - 60 minutes
 Dust concentration by volume - 0.88%

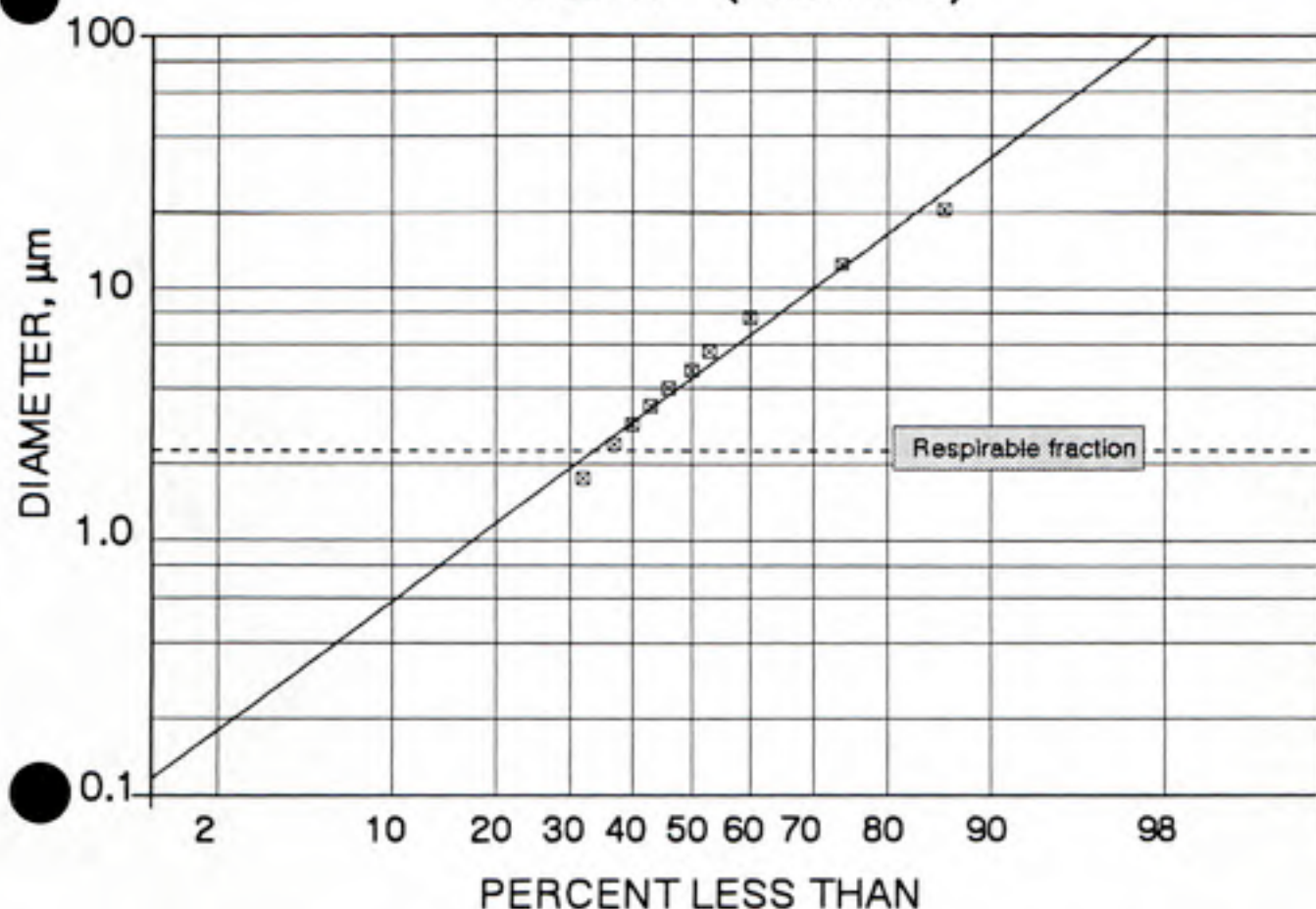
Particle Size Information

Respirable Fraction:
 Sedimentation - 12%
 Cyclone - n/a
 Manufacturer - 13%
 (Sized by L & N Microtrac Analyzer)

Sedimentation Data:
 Geometric mean - 6.08 microns
 Geometric std. deviation - 2.54

Figure 5. Particle size distribution by sedimentation method for Arizona Road Dust (Run #1).

CLAY (Run #1)

Physical Characteristics

Density - 2.65 gms/cm³
 Particle shape - spheres, plates and irregular
 Moisture - 1.55%

Particle Size Information

Respirable Fraction:
 Sedimentation - 32%
 Cyclone - 29%
 Manufacturer - n/a

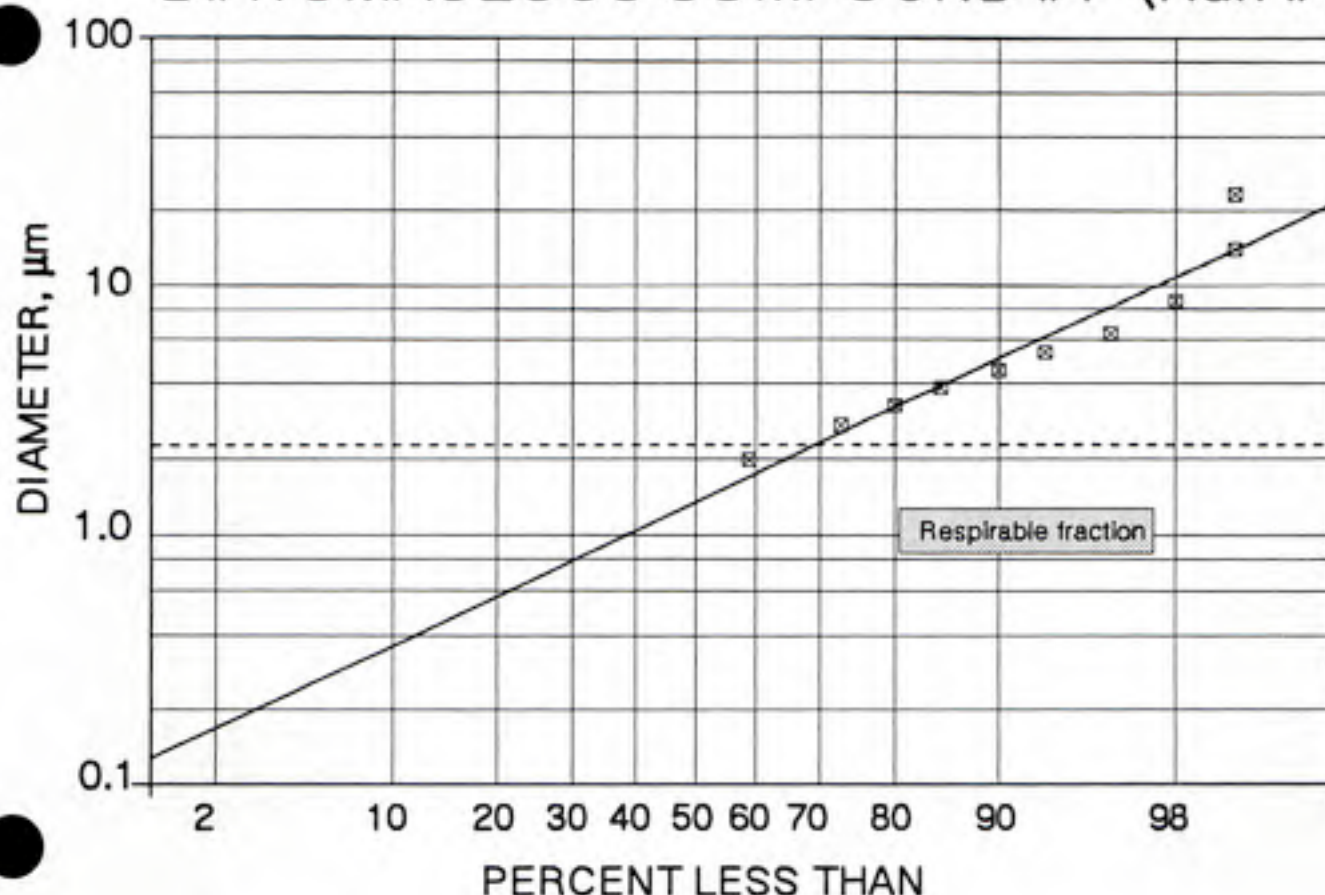
Sedimentation Conditions

Medium - water and ethyl alcohol
 Sampling height - 5 cm
 Sampling time - 60 minutes
 Dust concentration by volume - 1.18%

Sedimentation Data:
 Geometric mean - 4.41 microns
 Geometric std. deviation - 4.8

Figure 6. Particle size distribution by sedimentation method for Clay (Run #1).

DIATOMACEOUS COMPOUND #1 (Run #1)



Physical Characteristics

Density - 2.3 gms/cm³
 Particle shape - varied
 Moisture - 3.4%
 Bulking value - 17.5 lb/solid gal
 Porosity - diatoms contain many submicron pores
 % solubles - negligible

Sedimentation Conditions

Medium - water
 Sampling height - 5 cm
 Sampling time - 60 minutes
 Dust concentration by volume - 0.74%

Particle Size Information

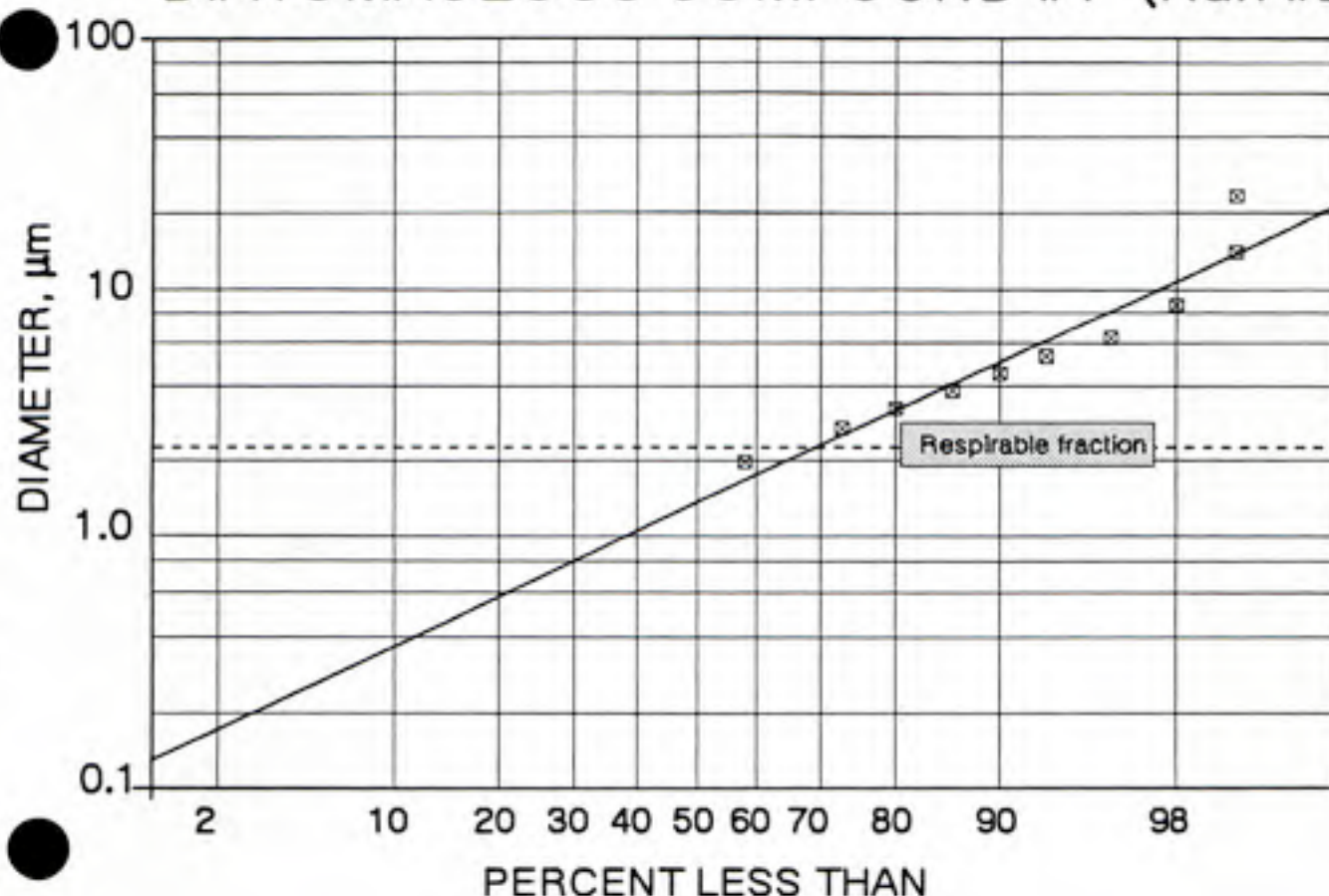
Respirable Fraction:
 Sedimentation - 65%
 Cyclone - 47%
 Manufacturer - 43%
 (Sized by Coulter Counter)

Sedimentation Data:
 Geometric mean - 1.35 microns
 Geometric std. deviation - 2.78

Manufacturer's data:
 Median particle size - 3.3 microns

Figure 7. Particle size distribution by sedimentation method for Diatomaceous Compound #1 (Run #1).

DIATOMACEOUS COMPOUND #1 (Run #2)



Physical Characteristics

Density - 2.3 gms/cm³
 Particle shape - varied
 Moisture - 3.4%
 Bulking value - 17.5 lb/solid gal
 Porosity - diatoms contain many submicron pores
 % solubles - negligible

Sedimentation Conditions

Medium - water
 Sampling height - 5 cm
 Sampling time - 60 minutes
 Dust concentration by volume - 0.94%

Particle Size Information

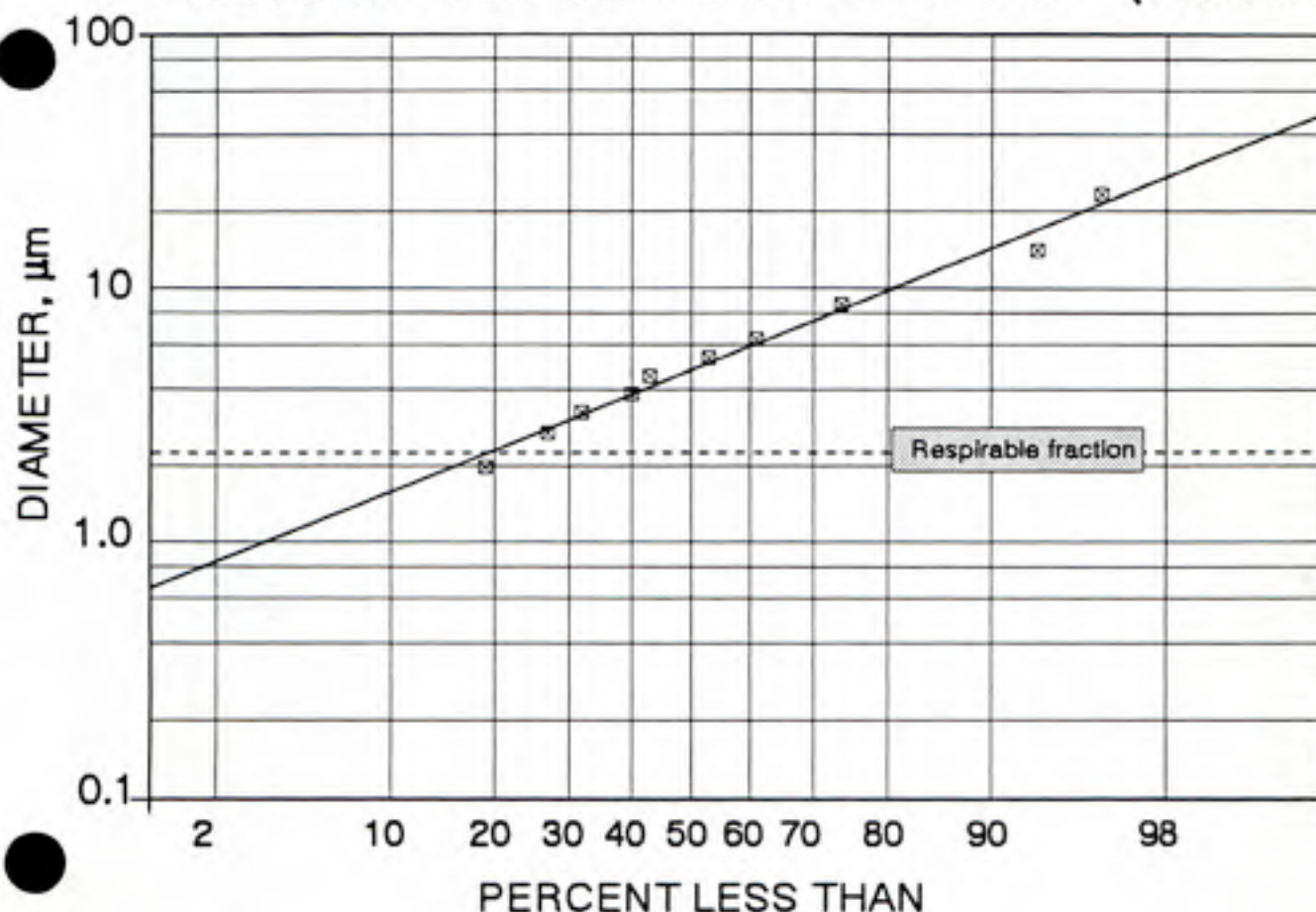
Respirable Fraction:
 Sedimentation - 65%
 Cyclone - 47%
 Manufacturer - 43%
 (Sized by Coulter Counter)

Sedimentation Data:
 Geometric mean - 1.36 microns
 Geometric std. deviation - 2.76

Manufacturer's data:
 Median particle size - 3.3 microns

Figure 8. Particle size distribution by sedimentation method for Diatomaceous Compound #1 (Run #2).

DIATOMACEOUS COMPOUND #2 (Run #1)



Physical Characteristics

Density - 2.3 gms/cm³
 Particle shape - varied
 Moisture - 3.3%
 Bulking value - 17.5 lb/solid gal
 Porosity - diatoms contain many submicron pores
 % solubles - negligible

Sedimentation Conditions

Medium - water
 Sampling height - 5 cm
 Sampling time - 60 minutes
 Dust concentration by volume - 0.38%

Particle Size Information

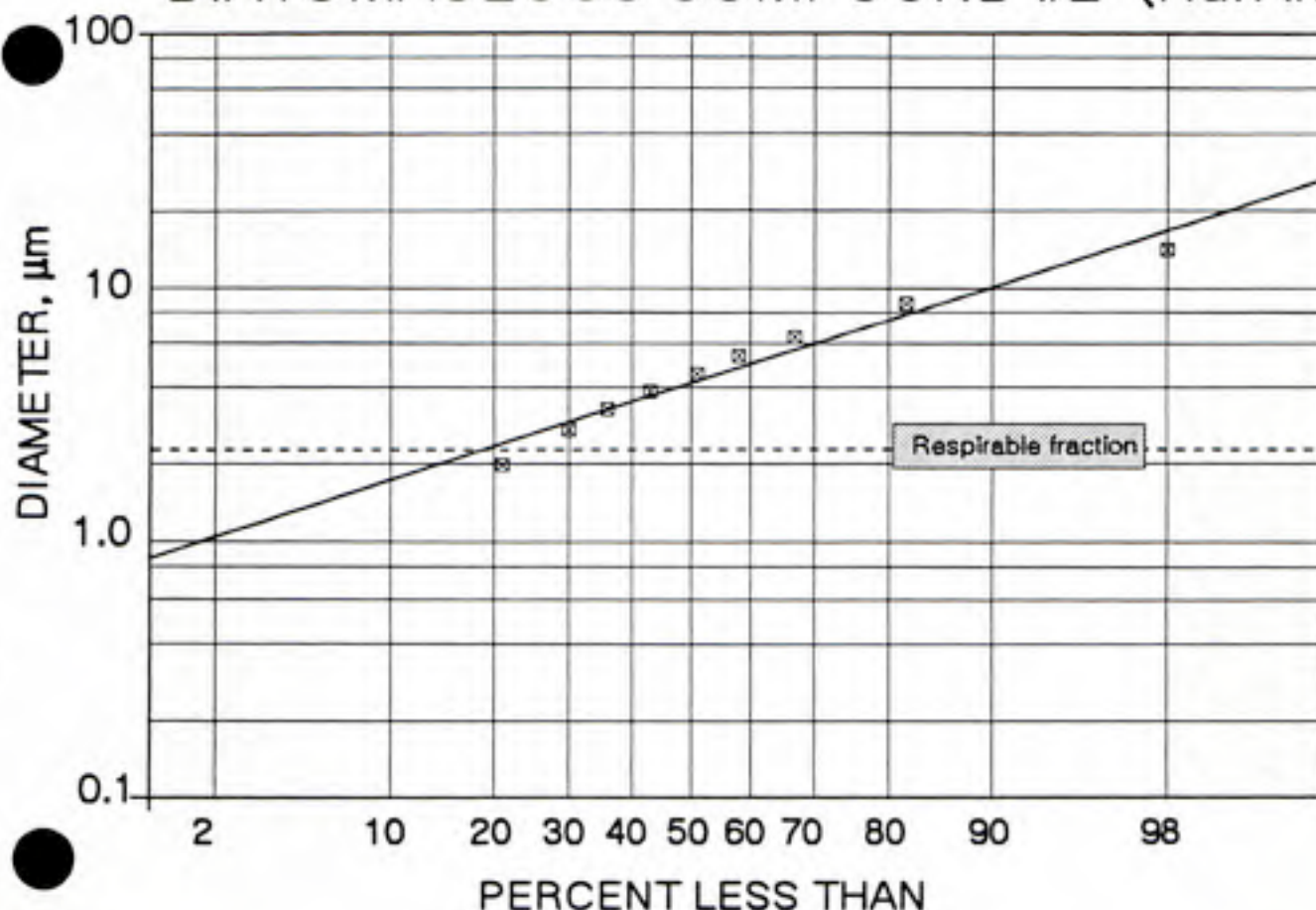
Respirable Fraction:
 Sedimentation - 18%
 Cyclone - 18%
 Manufacturer - 9%
 (Sized by Coulter Counter)

Sedimentation Data:
 Geometric mean - 4.76 microns
 Geometric std. deviation - 2.36

Manufacturer's data:
 Median particle size - 7.5 microns

Figure 9. Particle size distribution by sedimentation method for Diatomaceous Compound #2 (Run #1).

DIATOMACEOUS COMPOUND #2 (Run #2)



Physical Characteristics

Density - 2.3 gms/cm³
 Particle shape - varied
 Moisture - 3.3%
 Bulking value - 17.5 lb/solid gal
 Porosity - diatoms contain many submicron pores
 % solubles - negligible

Sedimentation Conditions

Medium - water
 Sampling height - 5 cm
 Sampling time - 60 minutes
 Dust concentration by volume - 0.38%

Particle Size Information

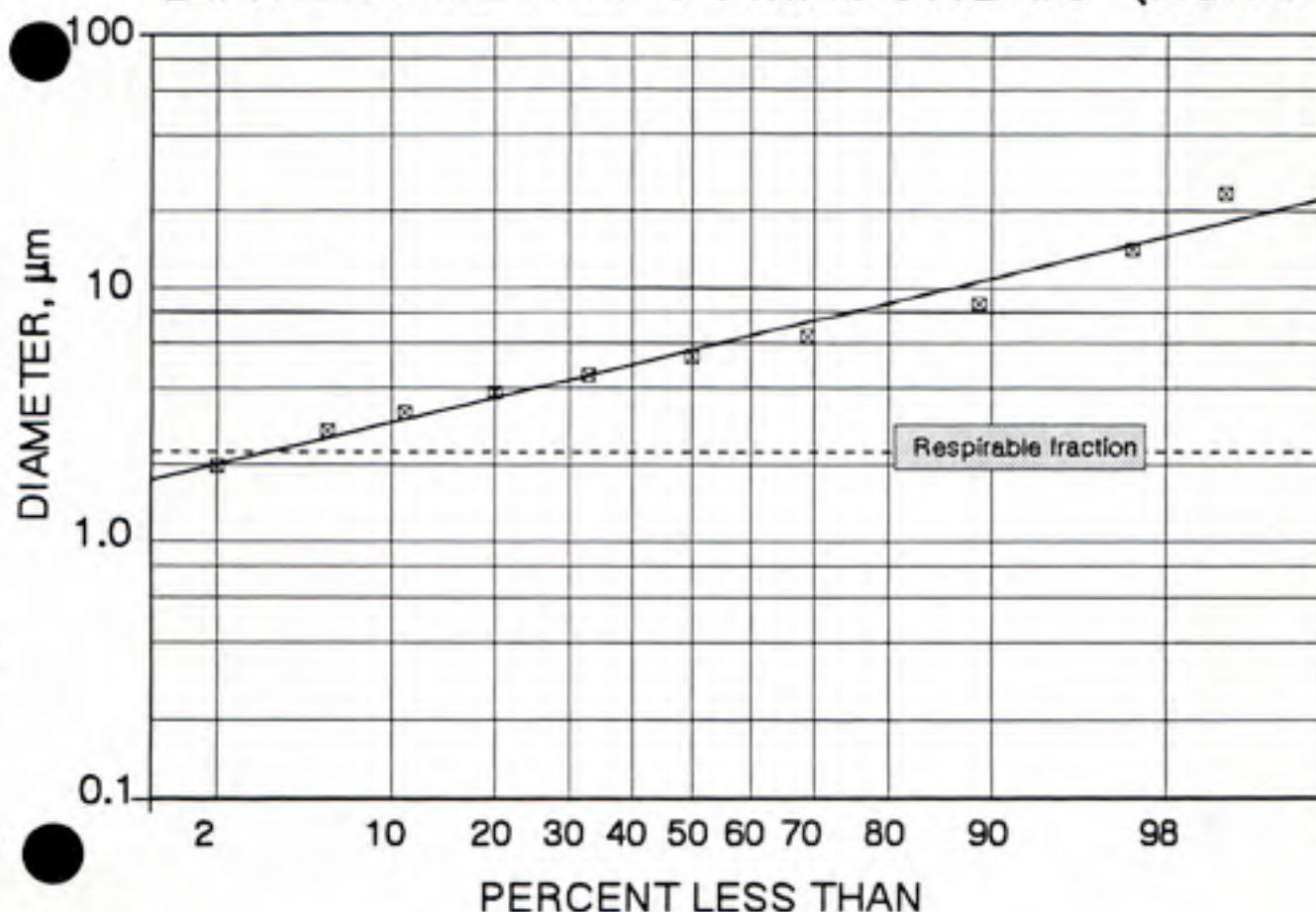
Respirable Fraction:
 Sedimentation - 17%
 Cyclone - 18%
 Manufacturer - 9%
 (Sized by Coulter Counter)

Sedimentation Data:
 Geometric mean - 4.16 microns
 Geometric std. deviation - 1.98

Manufacturer's data:
 Median particle size - 7.5 microns

Figure 10. Particle size distribution by sedimentation method for Diatomaceous Compound #2 (Run #2).

DIATOMACEOUS COMPOUND #3 (Run #1)



Physical Characteristics

Density - 2.3 gms/cm³
 Particle shape - varied
 Moisture - 0.2%
 Bulking value - 19.2 lb/solid gal
 Porosity - diatoms contain many submicron pores
 % solubles - negligible

Sedimentation Conditions

Medium - water
 Sampling height - 5 cm
 Sampling time - 60 minutes
 Dust concentration by volume - 0.62%

Particle Size Information

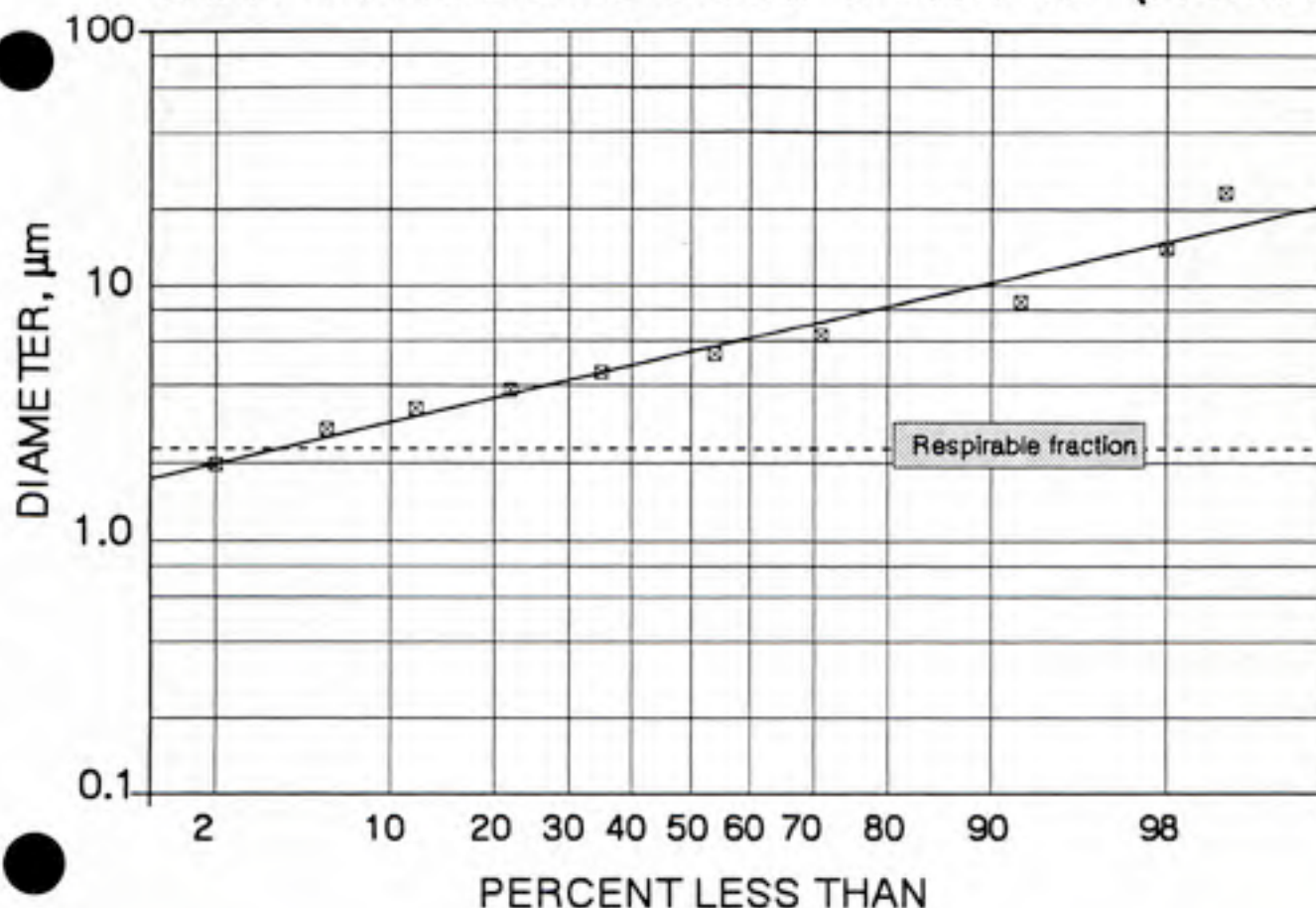
Respirable Fraction:
 Sedimentation - 5%
 Cyclone - 23%
 Manufacturer - 1%
 (Sized by Coulter Counter)

Sedimentation Data:
 Geometric mean - 5.77 microns
 Geometric std. deviation - 1.70

Manufacturer's data:
 Median particle size - 6.8 microns

Figure 11. Particle size distribution by sedimentation method for Diatomaceous Compound #3 (Run #1).

DIATOMACEOUS COMPOUND #3 (Run #2)



Physical Characteristics

Density - 2.3 gms/cm³
 Particle shape - varied
 Moisture - 0.2%
 Bulking value - 19.2 lb/solid gal
 Porosity - diatoms contain many submicron pores
 % solubles - negligible

Sedimentation Conditions

Medium - water
 Sampling height - 5 cm
 Sampling time - 60 minutes
 Dust concentration by volume - 0.9%

Particle Size Information

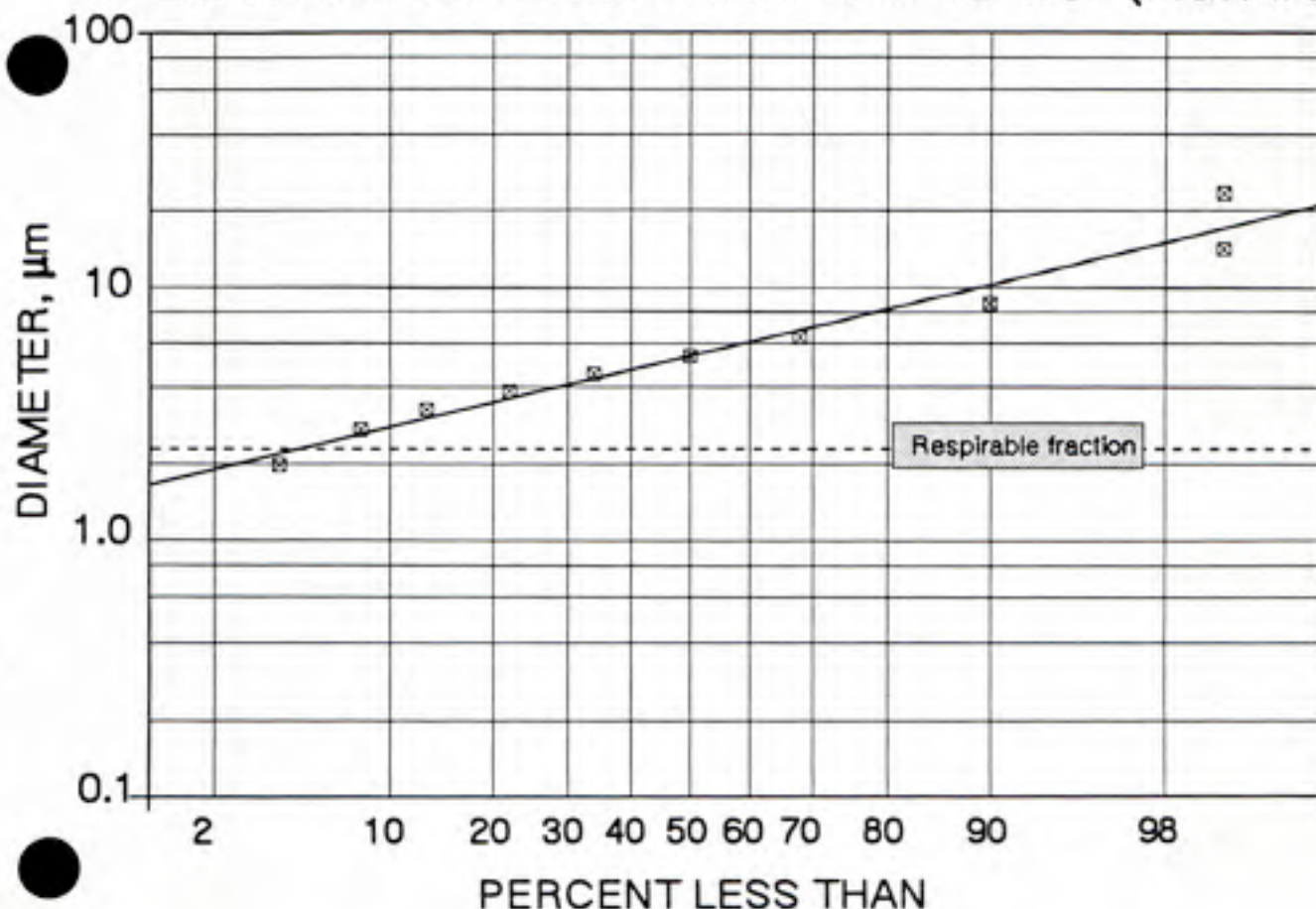
Respirable Fraction:
 Sedimentation - 5%
 Cyclone - 23%
 Manufacturer - 1%
 (Sized by Coulter Counter)

Sedimentation Data:
 Geometric mean - 5.45 microns
 Geometric std. deviation - 1.64

Manufacturer's data:
 Median particle size - 6.8 microns

Figure 12. Particle size distribution by sedimentation method for Diatomaceous Compound #3 (Run #2).

DIATOMACEOUS COMPOUND #3 (Run #3)



Physical Characteristics

Density - 2.3 gms/cm^3
 Particle shape - varied
 Moisture - 0.2%
 Bulking value - 19.2 lb/solid gal
 Porosity - diatoms contain many submicron pores
 % solubles - negligible

Sedimentation Conditions

Medium - water
 Sampling height - 5 cm
 Sampling time - 60 minutes
 Dust concentration by volume - 0.29%

Particle Size Information

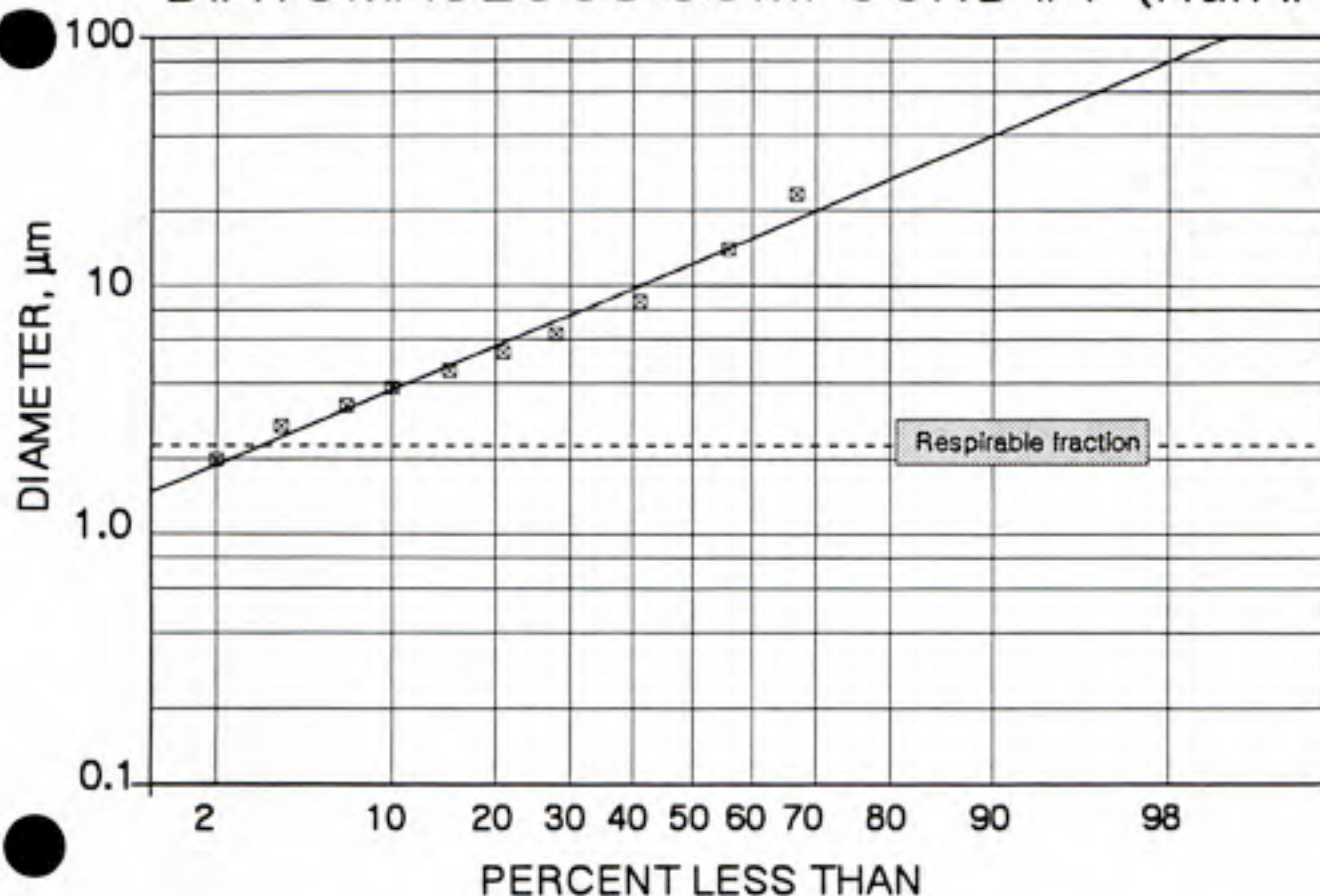
Respirable Fraction:
 Sedimentation - 4%
 Cyclone - 23%
 Manufacturer - 1%
 (Sized by Coulter Counter)

Sedimentation Data:
 Geometric mean - 5.34 microns
 Geometric std. deviation - 1.66

Manufacturer's data:
 Median particle size - 6.8 microns

Figure 13. Particle size distribution by sedimentation method for Diatomaceous Compound #3 (Run #3).

DIATOMACEOUS COMPOUND #4 (Run #1)



Physical Characteristics

Density - 2.3 gms/cm³
 Particle shape - varied
 Moisture - 0.5%
 % solubles - negligible
 Porosity - diatoms contain many submicron pores

Sedimentation Conditions

Medium - water
 Sampling height - 5 cm
 Sampling time - 60 minutes
 Dust concentration by volume - 1.05%

Particle Size Information

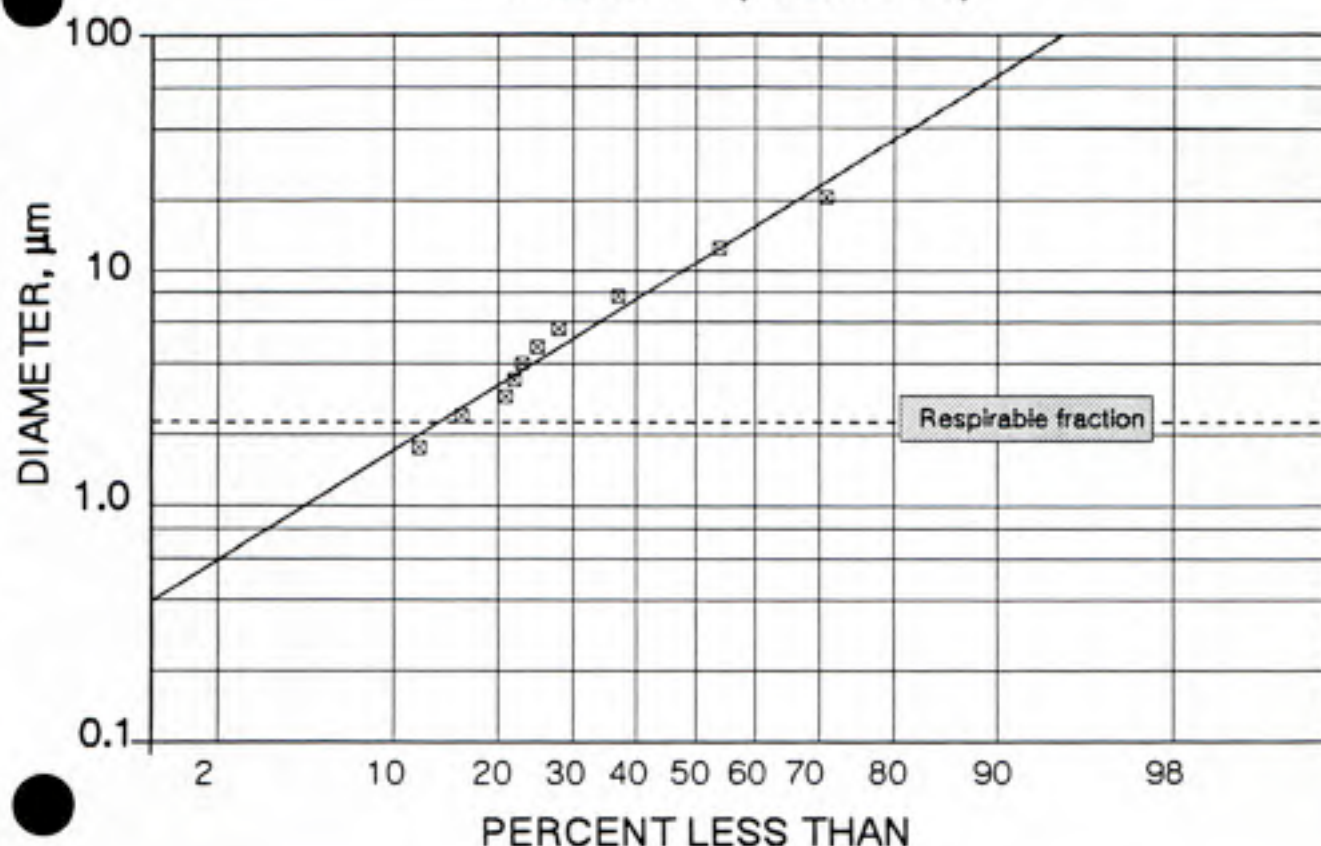
Respirable Fraction:
 Sedimentation - 5%
 Cyclone - 18%
 Manufacturer - 5%
 (Sized by Coulter Counter)

Sedimentation Data:
 Geometric mean - 12.32 microns
 Geometric std. deviation - 2.51

Manufacturer's data:
 Median particle size - 15 microns

Figure 14. Particle size distribution by sedimentation method for Diatomaceous Compound #4 (Run #1).

FLINT (Run #1)



Physical Characteristics

Density - 2.65 gms/cm³
 Particle shape - spheres, plates and irregular
 Moisture - 0.04%

Particle Size Information

Respirable Fraction:
 Sedimentation - 13%
 Cyclone - 39%
 Manufacturer - n/a

Sedimentation Conditions

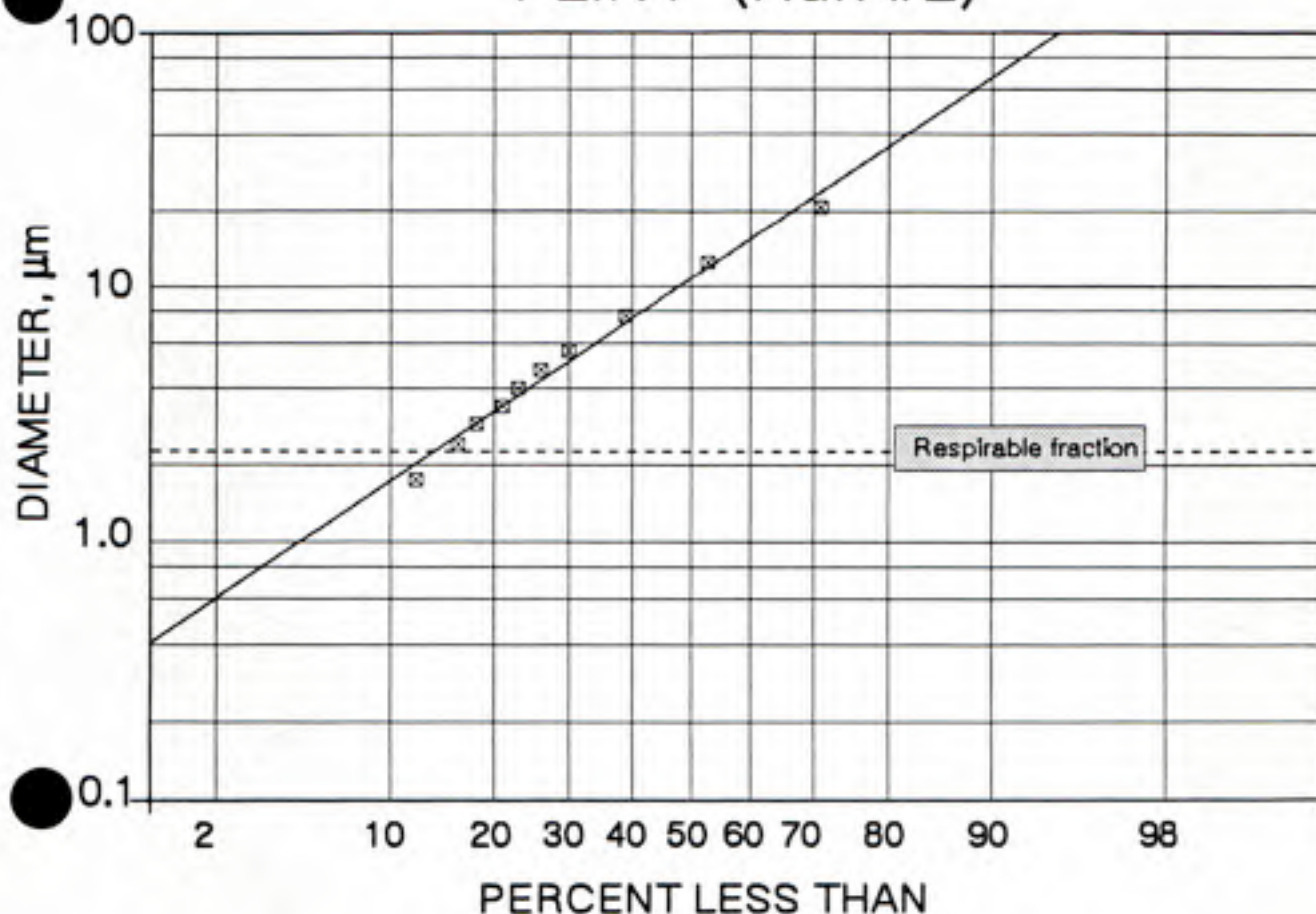
Medium - water
 Sampling height - 5 cm
 Sampling time - 60 minutes
 Dust concentration by volume - 0.49%

Sedimentation Data:
 Geometric mean - 10.77 microns
 Geometric std. deviation - 4.16

Note: Dust cloud that developed while handling resulted in significant loss of sample. Probable resulting effect was underestimation of respirable fraction.

Figure 15. Particle size distribution by sedimentation method for Flint (Run #1).

FLINT (Run #2)



Physical Characteristics

Density - 2.65 gms/cm³
 Particle shape - spheres, plates and irregular
 Moisture - 0.04%

Particle Size Information

Respirable Fraction:
 Sedimentation - 13%
 Cyclone - 39%
 Manufacturer - n/a

Sedimentation Conditions

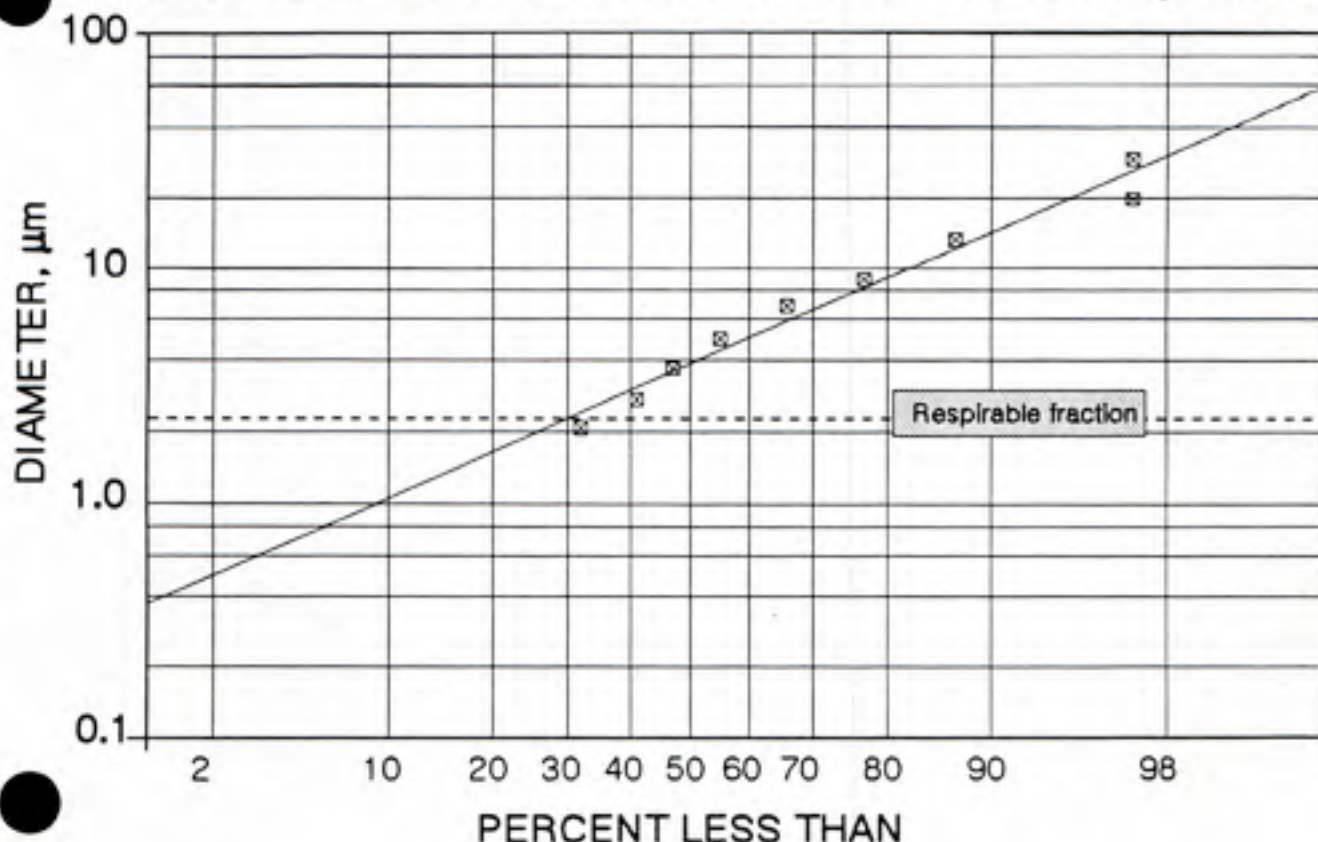
Medium - water and ethyl alcohol
 Sampling height - 5 cm
 Sampling time - 60 minutes
 Dust concentration by volume - 0.92%

Sedimentation Data:
 Geometric mean - 10.75 microns
 Geometric std. deviation - 4.12

Note: Dust cloud that developed while handling resulted in significant loss of sample. Probable resulting effect was underestimation of respirable fraction.

Figure 16. Particle size distribution by sedimentation method for Flint (Run #2).

MICRONIZED AMORPHOUS SILICA (Run #1)



Physical Characteristics

Density - 2.65 gms/cm^3
 Particle shape - Mostly spheres
 Moisture - 0.25%
 Bulking Value - 22.07 lbs/solid gal
 Apparent Density (Scott Volumeter) - 24 lbs/ft³

Sedimentation Conditions

Medium - Water
 Sampling height - 5 cm
 Sampling time - 60 minutes
 Dust concentration by volume - 1.06%

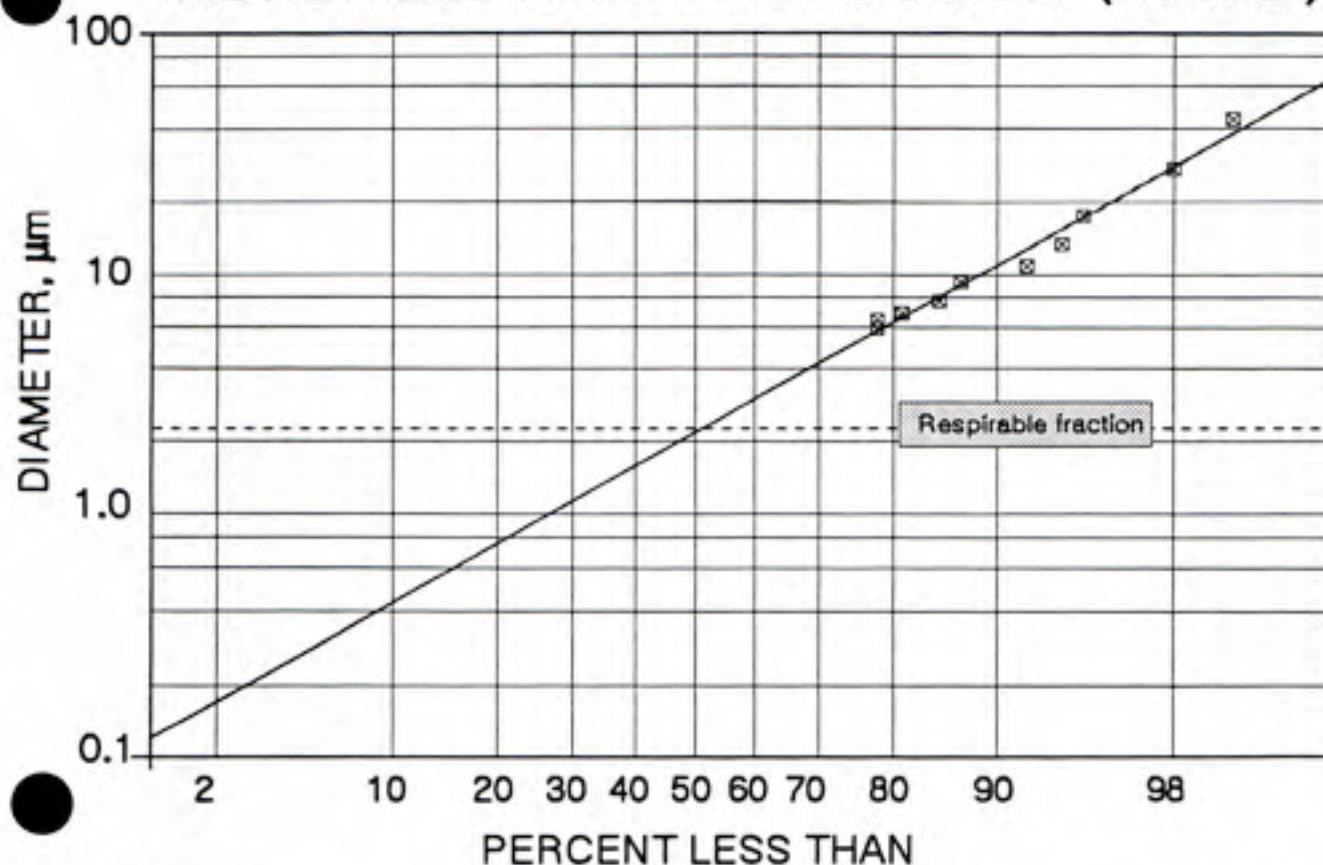
Particle Size Information

Respirable Fraction:
 Sedimentation - 32%
 Cyclone - 29%
 Manufacturer
 (Method unknown) - 35%

Sedimentation Data:
 Geometric mean - 3.88 microns
 Geometric std. deviation - 2.75

Figure 17. Particle size distribution by sedimentation method for Micronized Amorphous Silica (Run #1).

MICRONIZED AMORPHOUS SILICA (Run #2) *



* Note: Sampling Height = 20 Centimeters . The method is not considered applicable to the particle sizes sampled under these conditions.

Physical Characteristics

Density - 2.65 gms/cm³
 Particle shape - Mostly spheres
 Moisture - 0.25%
 Bulking Value - 22.07 lbs/solid gal
 Apparent Density (Scott Volumeter) - 24 lbs/ft³

Particle Size Information

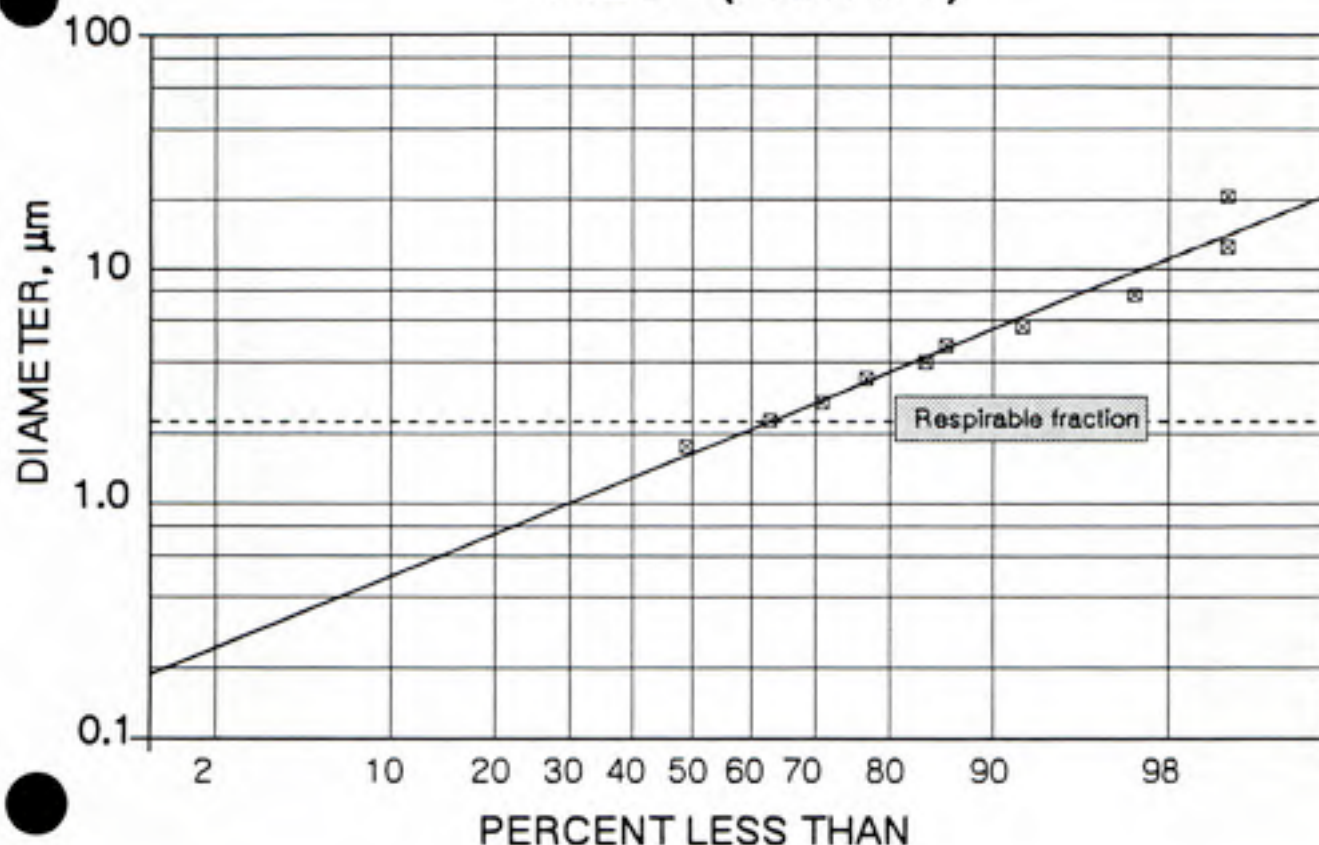
Respirable Fraction:
 Sedimentation - 50%
 Cyclone - 29%
 Manufacturer
 (Method unknown) - 35%

Sedimentation Conditions

Medium - water
 Sampling height - 20 cm
 Sampling time - 60 minutes
 Dust concentration by volume - 1.06%

Figure 18. Particle size distribution by sedimentation method for Micronized Amorphous Silica (Run #2).

TALC (Run #1)

Physical Characteristics

Density - 2.65 gms/cm
 Particle shape - plates and irregular
 Moisture - 0.5%
 Bulking value - 22.5 lb/solid gal
 % solubles - < 1%

Particle Size Information

Respirable Fraction:
 Sedimentation - 62%
 Cyclone - n/a
 Manufacturer - 35%
 (Method unknown)

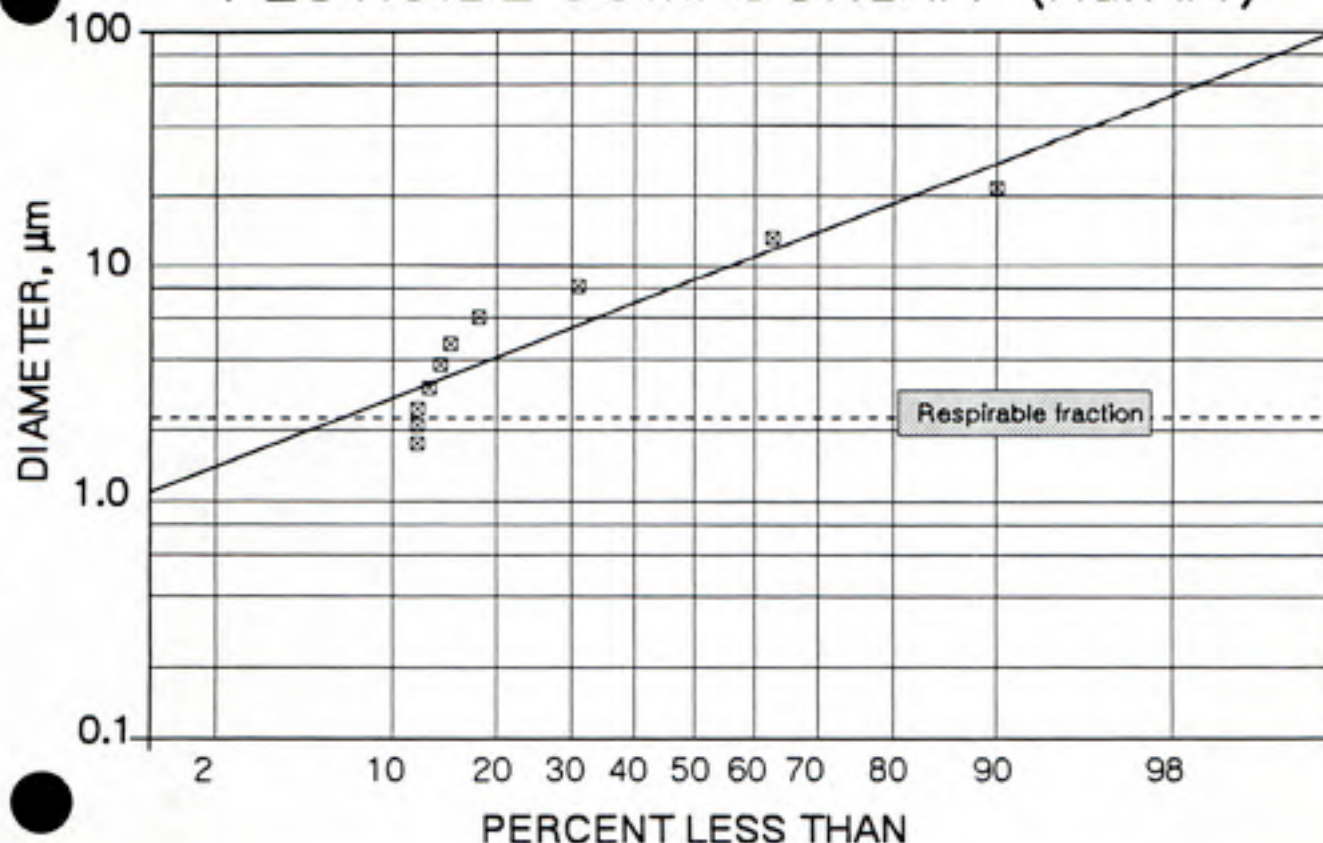
Sedimentation Conditions

Medium - water
 Sampling height - 5 cm
 Sampling time - 60 minutes
 Dust concentration by volume - 0.86%

Sedimentation Data:
 Geometric mean - 1.65 microns
 Geometric std. deviation - 2.55

Figure 19. Particle size distribution by sedimentation method for Talc (Run #1).

PESTICIDE COMPOUND #1 (Run #1)



Physical Characteristics

Density - 2.65 gms/cm³
Particle shape - irregular

Particle Size Information

Respirable Fraction:
Sedimentation - 12%
Cyclone - 12%
Manufacturer - n/a

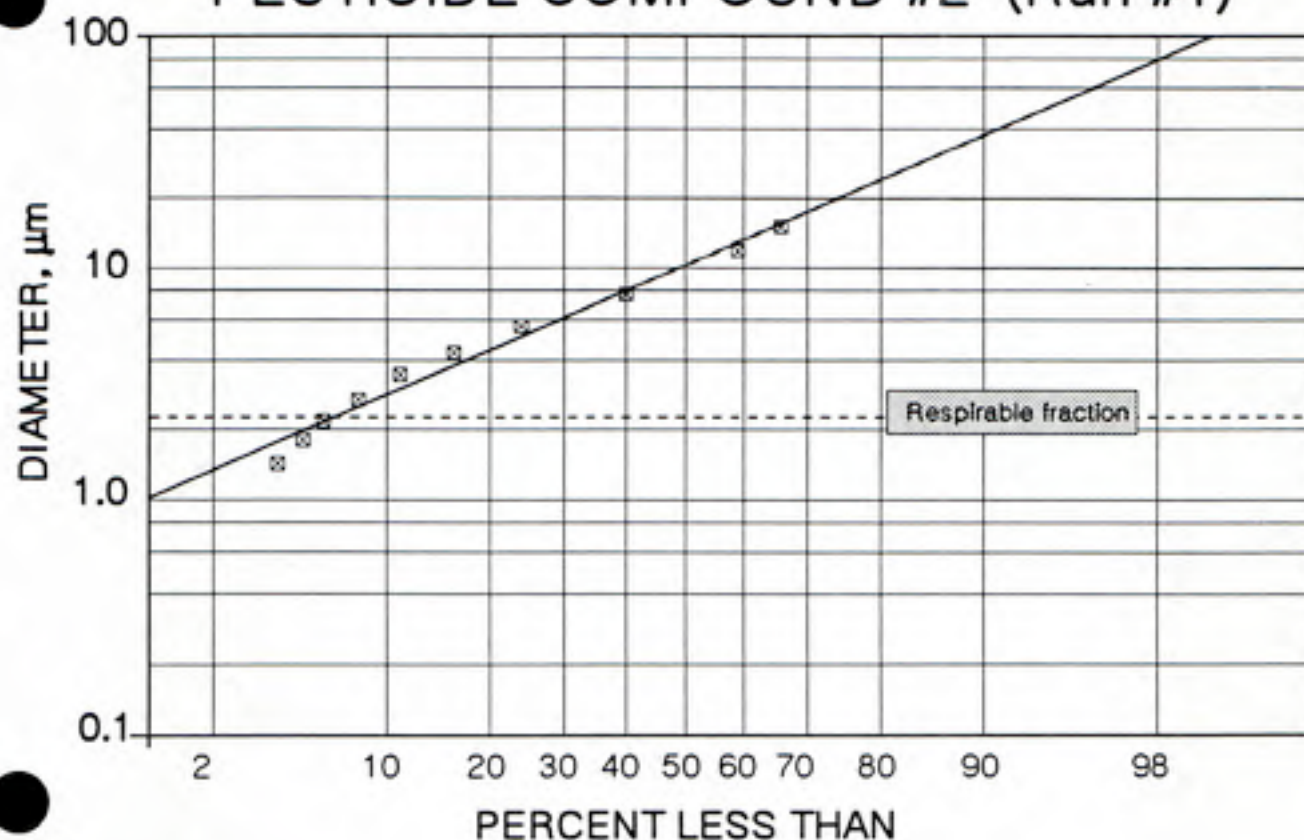
Sedimentation Conditions

Medium - Water
Sampling height - 5 cm
Sampling time - 90 minutes
Dust concentration by volume - 0.24%

Sedimentation Data:
Geometric mean - 8.73 microns
Geometric std. deviation - 2.46

Figure 20. Particle size distribution by sedimentation method for Pesticide Compound #1 (Run #1).

PESTICIDE COMPOUND #2 (Run #1)



Physical Characteristics

Density - 2.65 gms/cm^3
 Particle shape - irregular

Particle Size Information

Respirable Fraction:
 Sedimentation - 6%
 Cyclone - 6%
 Manufacturer - n/a

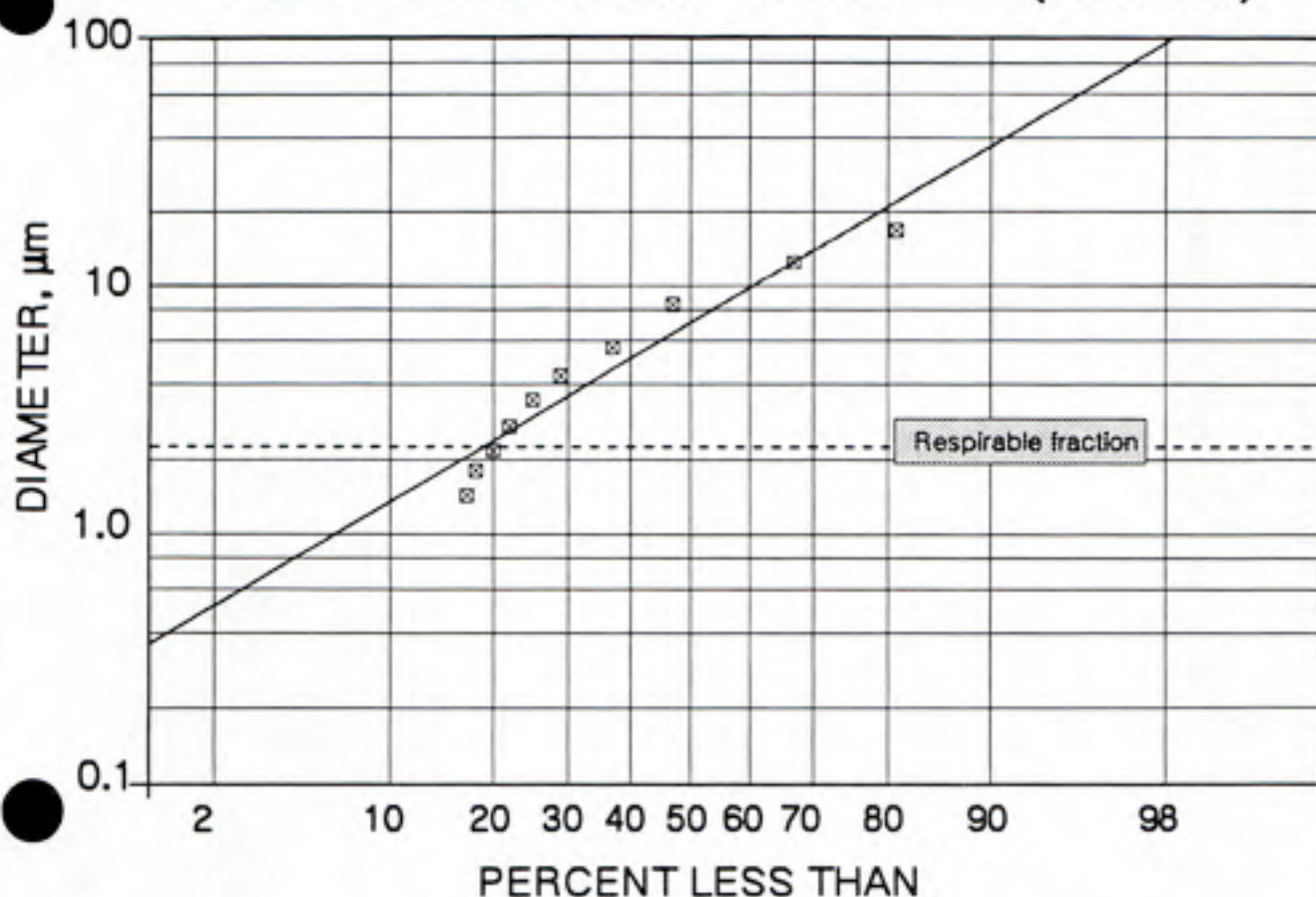
Sedimentation Conditions

Medium - Water
 Sampling height - 5 cm
 Sampling time - 90 minutes
 Dust concentration by volume - 0.52%

Sedimentation Data:
 Geometric mean - 10.3 microns
 Geometric std. deviation - 2.72

Figure 21. Particle size distribution by sedimentation method for Pesticide Compound #2 (Run #1).

PESTICIDE COMPOUND #3 (Run #1)



Physical Characteristics

Density - 2.65 gms/cm³
 Particle shape - irregular

Particle Size Information

Respirable Fraction:
 Sedimentation - 17%
 Cyclone - 15%
 Manufacturer - n/a

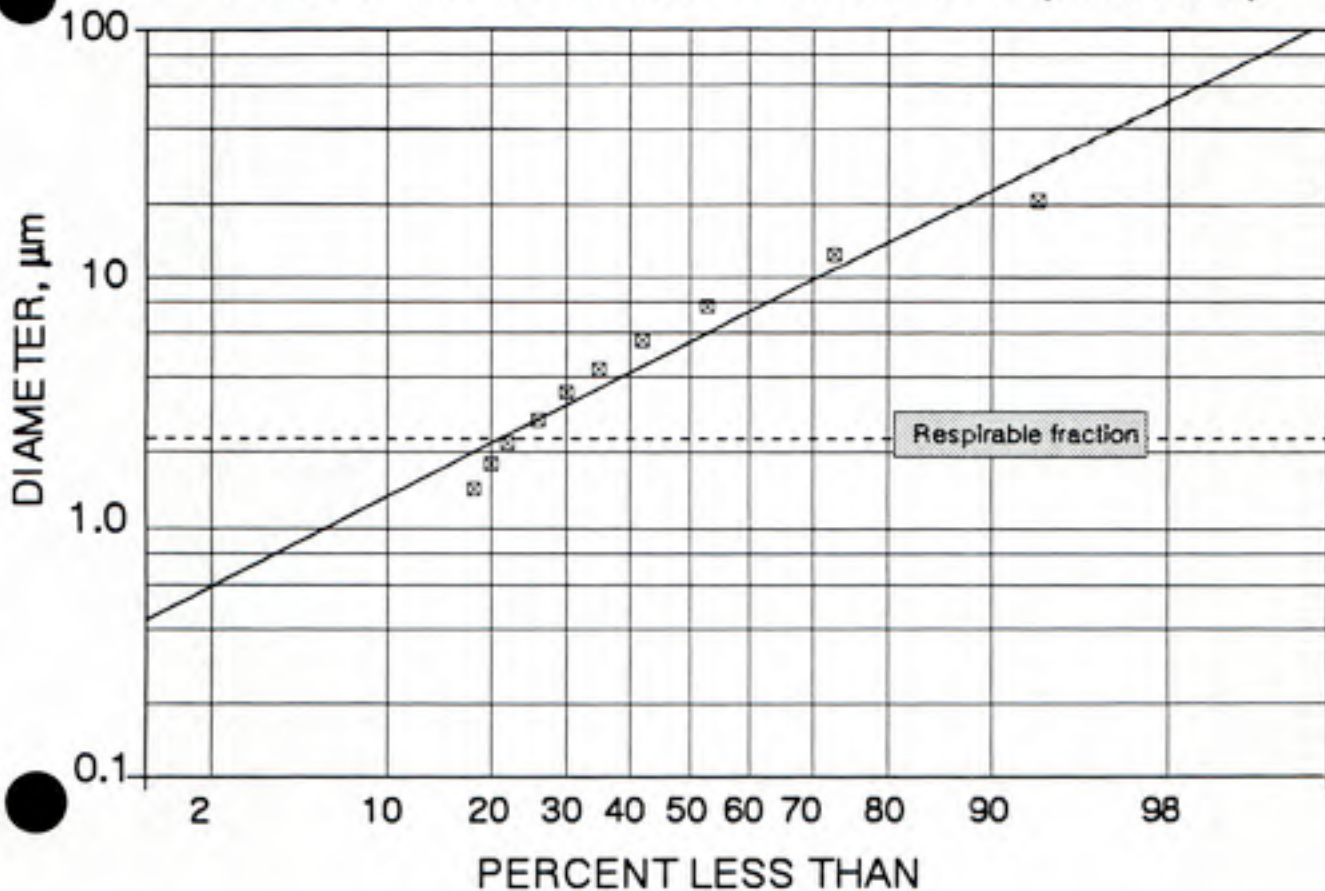
Sedimentation Conditions

Medium - Water
 Sampling height - 5 cm
 Sampling time - 90 minutes
 Dust concentration by volume - 0.55%

Sedimentation Data:
 Geometric mean - 7.07 microns
 Geometric std. deviation - 3.62

Figure 22. Particle size distribution by sedimentation method for Pesticide Compound #3 (Run #1).

PESTICIDE COMPOUND #4 (Run #1)



Physical Characteristics

Density - 2.65 gms/cm³
Particle shape - Irregular

Particle Size Information

Respirable Fraction:
Sedimentation - 22%
Cyclone - 21%
Manufacturer - n/a

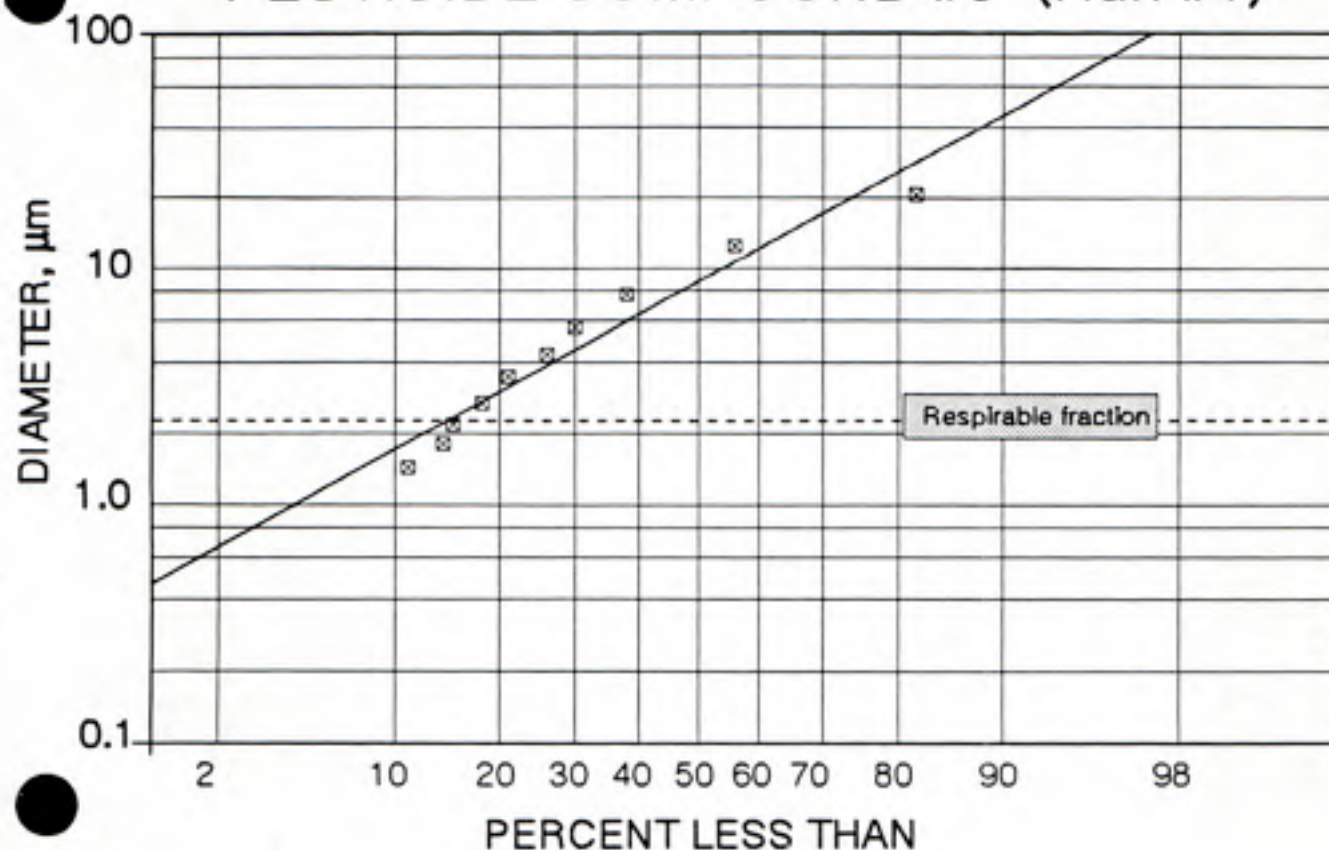
Sedimentation Conditions

Medium - water
Sampling height - 5 cm
Sampling time - 90 minutes
Dust concentration by volume - 0.36%

Sedimentation Data:
Geometric mean - 5.52 microns
Geometric std. deviation - 3.0

Figure 23. Particle size distribution by sedimentation method for Pesticide Compound #4 (Run #1).

PESTICIDE COMPOUND #5 (Run #1)



Physical Characteristics

Density - 2.65 gms/cm³
Particle shape - irregular

Particle Size Information

Respirable Fraction:
Sedimentation - 12%
Cyclone - 18%
Manufacturer - n/a

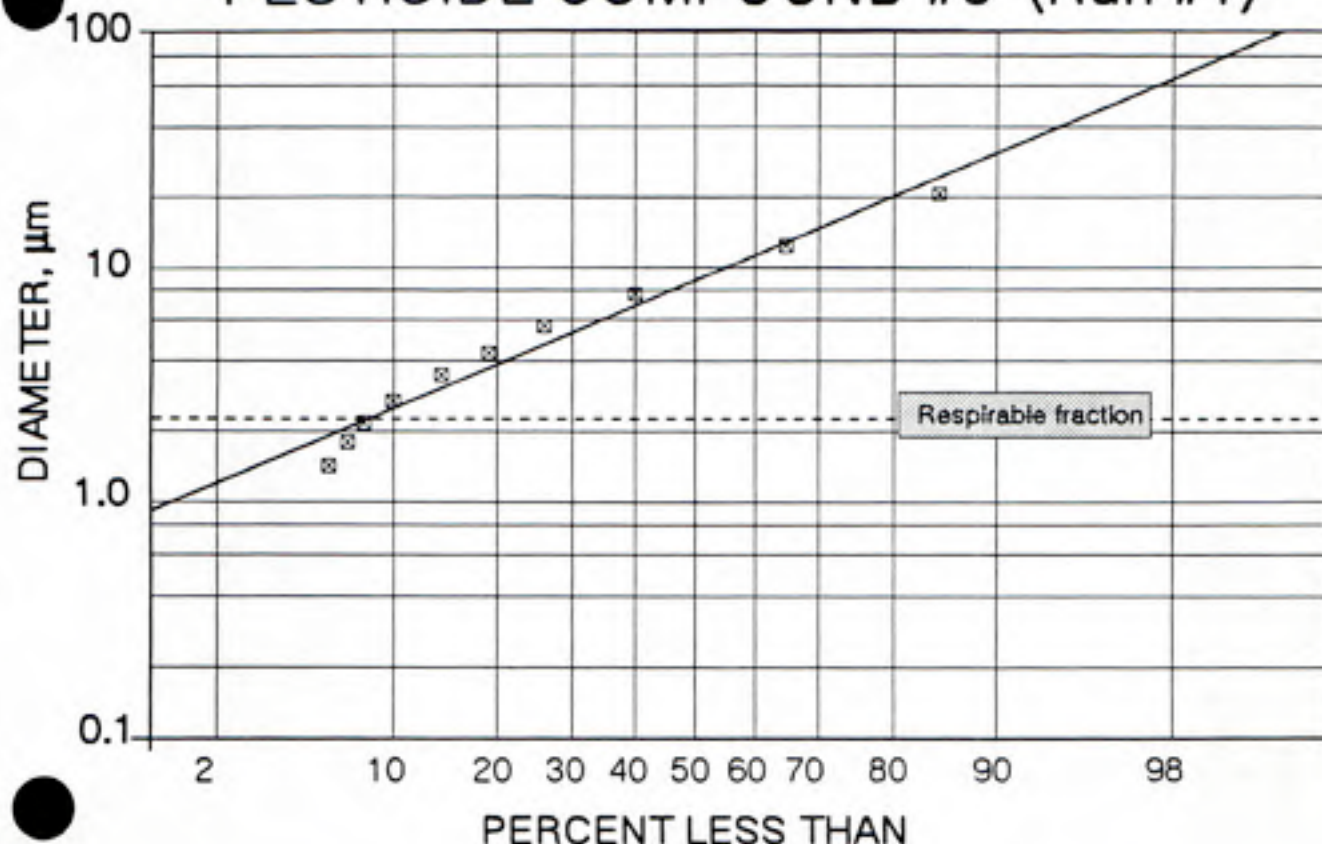
Sedimentation Conditions

Medium - water
Sampling height - 5 cm
Sampling time - 90 minutes
Dust concentration by volume - 0.44%

Sedimentation Data:
Geometric mean - 8.78 microns
Geometric std. deviation - 3.56

Figure 24. Particle size distribution by sedimentation method for Pesticide Compound #5 (Run #1).

PESTICIDE COMPOUND #6 (Run #1)



Physical Characteristics

Density - 2.65 gms/cm³
 Particle shape - irregular

Particle Size Information

Respirable Fraction:
 Sedimentation - 9%
 Cyclone - 13%
 Manufacturer - n/a

Sedimentation Conditions

Medium - Water
 Sampling height - 5 cm
 Sampling time - 90 minutes
 Dust concentration by volume - 0.44%

Sedimentation Data:
 Geometric mean - 8.80 microns
 Geometric std. deviation - 2.65

Figure 25. Particle size distribution by sedimentation method for Pesticide Compound #6 (Run #1).

progression in particle size (D) as calculated by Stokes' equation and shown by equation #6 (4,9).

Cyclone Data - Respirable Fraction Measurements

Respirable fraction measurements were performed for a total of 13 different dusts using 10-mm nylon cyclones and 37-mm cassettes loaded with PVC filters and operated at 1.7 LPM. Each run consisted of three two-stage respirable samplers and one total dust sampler. The amount of dust injected into the chamber ranged from 0.77 to 14 grams of material, and sampling time varied from 5 to 21 minutes. The respirable dust fraction is represented by the ratio of the amount of dust collected on the filter following the cyclone precollector to the amount of dust collected on the open-face filter. Six or half of the test dusts' respirable samples were sent to outside laboratories where x-ray diffraction analysis was employed for quantification of crystalline silica. The results of the cyclone runs are summarized in Table V.

For each dust, a minimum of three separate loading-sampling runs was conducted. In these tests an attempt was made to vary dust concentration so that 1) there would be a better chance of collecting more than the minimum detectable weight of free silica on a filter, and 2) the assumption that the predicted respirable fraction was independent of the amount of dust injected into the chamber would be demonstrated. Figures 26-28 show plots of the respirable fraction (y) as a function of the amount of dust injected (x) into the chamber. Although a regression line fit to the

Table V-A.
Cyclone Respirable Fraction Data

Dust	Run	Dust Mass (gms)	Sampling Time (min)	Respirable Fraction(%)			Average Respirable Fraction(%)	Total Average Respirable Fraction(%) From All Runs
Flint	1	5.72	14.0	22.5	17.7	49.7	30.0	39.0%
	2	3.11	10.0	46.2	50.2	48.2	48.2	
	3	2.41	10.0	51.3	30.7	34.8	38.9	
Clay	1	7.46	7.0	18.0	20.8	31.6	23.5	29.1%
	2	3.73	8.0	22.9	26.0	41.2	30.0	
	3	4.52	10.0	24.2	43.8	33.5	33.9	
Diatomaceous Compound #1 **	1	1.61	5.0	35.5	54.2	50.0	45.9	47.2%
	2	0.49	10.0	36.2	58.4	56.3	50.3	
	3	0.88	6.0	55.9	58.2	22.2	45.4	
Diatomaceous Compound #2	1	0.77	10.0	18.2	16.7	12.9	15.9	17.9%
	2	1.36	10.0	19.0	20.7	19.8	19.8	
	3	1.18	10.0	15.6	20.3	52.5*	17.9	
Diatomaceous Compound #3 **	1	2.33	7.0	33.7	2.9	43.5	26.7	23.0%
	2	2.09	12.0	18.5	14.5	24.8	19.3	
Diatomaceous Compound #4 **	1	1.69	7.0	3.1	26.1	31.9	20.4	18.3%
	2	2.68	7.0	17.7	31.1	1.0	24.4	
	3	2.36	10.5	18.2	11.1	2.0	10.4	
	4	2.55	10.3	13.2	35.4*	5.2	17.9	

* BGI Cyclone - Data not included in average.

** Particles contained high charge which caused significant particle agglomeration.

Table V-B.

Cyclone Respirable Fraction Data

Micronized Amorphous Silica	1	2.00	10.0	28.8	27.3	26.1	27.4	29.2%
	2	6.02	20.0	35.1	14.1	20.3	23.2	
	3	3.99	20.0	25.7	5.6	29.8	20.4	
	4	6.00	10.0	44.5	27.8		36.1	
	5	6.59	10.0	66.9	32.9	43.8	47.9	
	6	4.23	10.0	39.5	39.0	26.0	34.8	
	7	1.60	10.0	19.4	22.3	3.0	14.9	
Pesticide Compound #1	1	10.0	15.0		10.4			12.0%
	2	8.0	21.0		12.3			
Pesticide Compound #2	1	7.0	16.0		3.0			6.0%
	2	9.7	16.0		7.5			
	3	9.4	16.0		8.4			
Pesticide Compound #3	1	8.9	20.0		16.0			14.9%
	2	7.1	21.0		20.0			
	3	4.2	20.0		8.6			
Pesticide Compound #4	1	7.0	16.0		13.1			20.6%
	2	9.7	16.0		21.5			
	3	9.4	16.0		27.2			
Pesticide Compound #5	1	14.0	19.0		30.6			18.2%
	2	7.7	13.0		12.0			
	3	4.7	12.0		12.0			
Pesticide Compound #6	1	7.4	15.0		19.1			13.1%
	2	12.3	10.0		8.3			
	3	3.2	16.0		12.0			

* BGI Cyclone - Data not included in average.

** Particles contained high charge which caused significant particle agglomeration.

CHAMBER LOADING MEASUREMENTS CLAY

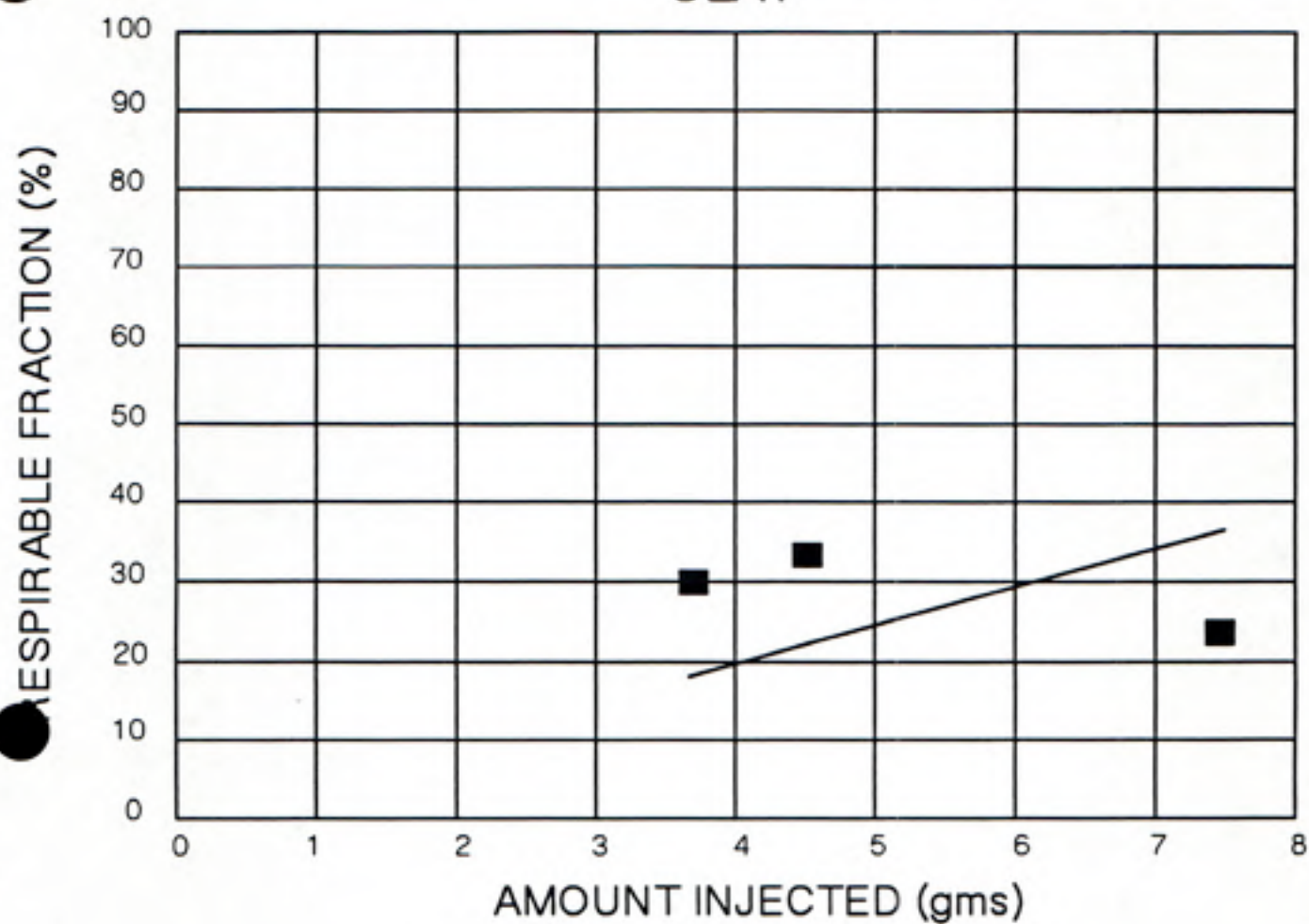


Figure 26. Respirable fraction as a function of chamber loading for Clay.

DIATOMACEOUS COMPOUND #2

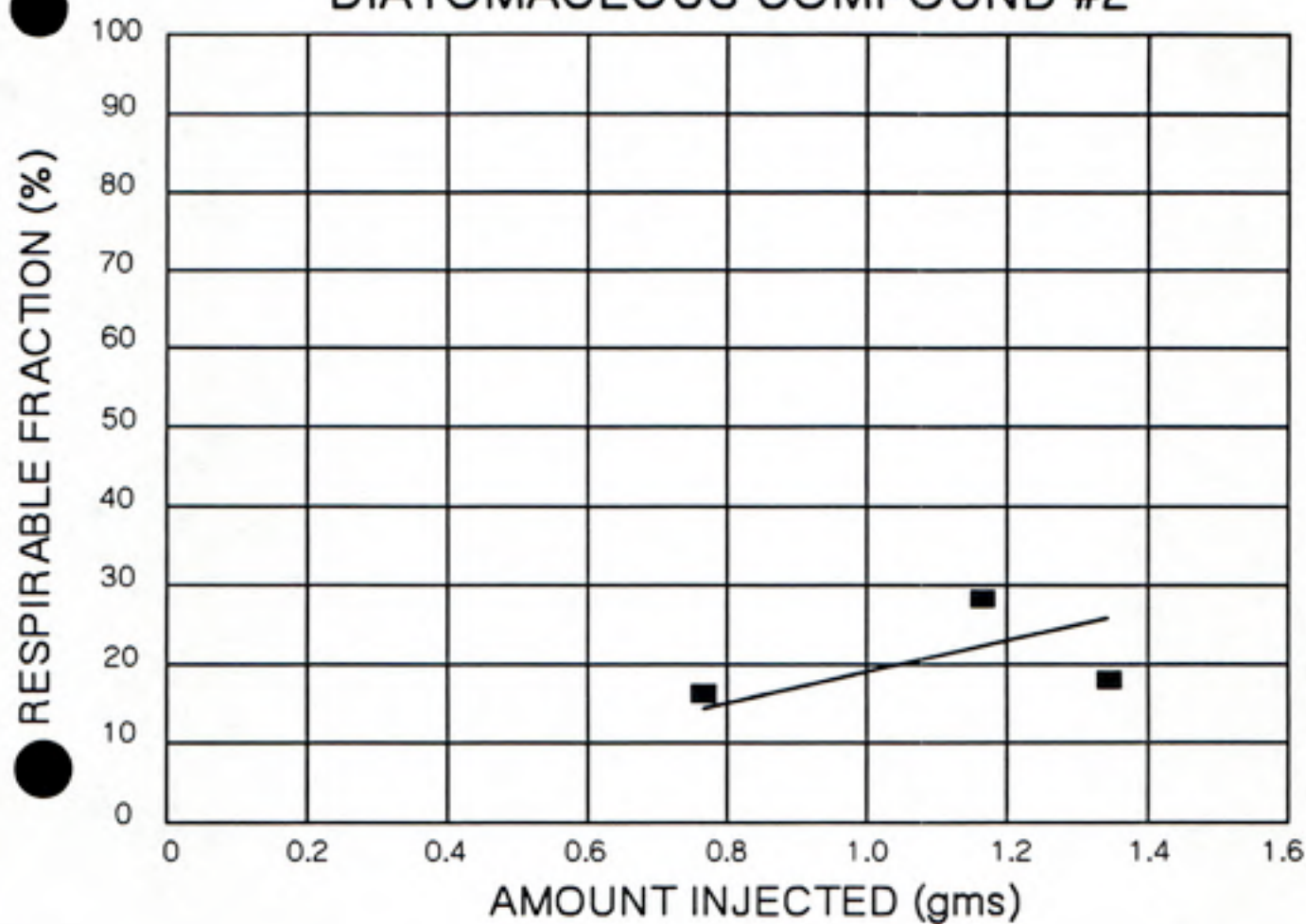


Figure 27. Respirable fraction as a function of chamber loading for Diatomaceous Compound #2.

CHAMBER LOADING MEASUREMENTS MICRONIZED AMORPHOUS SILICA

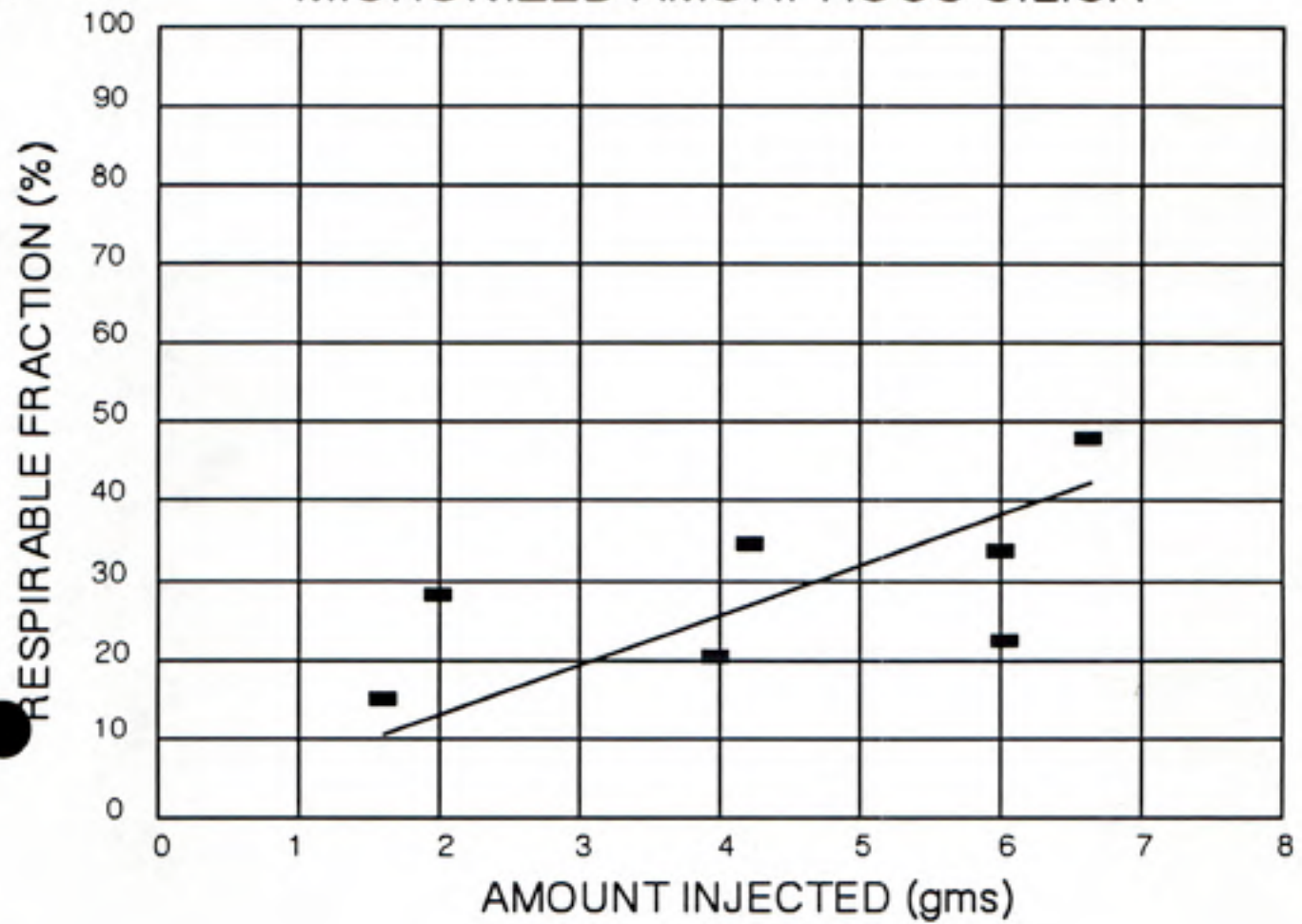


Figure 28. Respirable fraction as a function of chamber loading for Micronized Amorphous Silica.

data indicates in each case a positive slope (i.e., some sort of linear relationship between x and y), in actuality the r^2 values for all three tests shown were less than 0.2, indicating a very low probability of correlation between the predicted respirable fraction and the amount of dust injected into the chamber. R^2 values, which measure the strength of the linear relationship between x and y, range from 0 to 1. If $r^2 = 0$, there is no linear relationship between x and y. If $r^2 = 1$, there is a perfect linear relationship between x and y.

Respirable fraction measurements showed high degrees of variability within the test chamber for some dusts, while for others agreement between the three runs was exceptionally close. For the 13 dusts sampled, variability in the respirable fraction measurements ranged from very low ($\pm 1.9\%$) as was the case with pesticide compound #1, to high ($\pm 33\%$) as shown by the micronized amorphous silica dust. Variability for 10 of the 13 dusts was within the limits of $\pm 15\%$.

Variability between the three separate cyclones' measurements within an individual run was also inconsistent. For example, Run #2 for diatomaceous compound #2 showed excellent agreement within $\pm 2\%$ between the three cyclones' respirable fraction measurements, while flint showed variability of $\pm 32\%$ between the three measurements.

Attempts to target the source of this variability were greatly complicated by the inability to distinguish effects caused by the chamber and those caused by the cyclones. In other words, were the variable results caused by a nonuniform dust cloud within the chamber, or were they caused by varying levels in performance between the 3 cyclones? The effect was investigated with several dusts by alternating the cyclones in a clockwise fashion from one run to the next. For example, in Run #2, cyclone #1 would be moved to cyclone #2's previous position, cyclone #2 moved to cyclone #3's previous position, and cyclone #3 to cyclone #1's previous position. For these studies, individual cyclones failed to predict identical respirable fractions from one chamber location to another. This seemed to show that the variability was within the dust cloud rather than the cyclone's performance. This observation was further supported by data which showed respirable results that were repeated from one run to the next but by different cyclones. For example, on Run #1 cyclone #1 predicted a respirable fraction of 15% and cyclone #2 predicted a respirable fraction of 24%, and on Run #2 vice versa. Although these trends support the theory of nonuniformity within the dust cloud, a definitive conclusion could not be reached because of potential confounding factors such as pump fluctuations, leaky cyclones, aggregation of highly charged particles, etc.

Comparison of Sedimentation, Cyclone
and Manufacturers' Respirable Fraction Data

Table VI shows a comparison between the sedimentation method's respirable predictions and those of the cyclone for each dust. The table also includes respirable data provided by the manufacturer. Cyclone values represent an average value of all runs conducted for a particular dust.

For the pesticides, the sedimentation data are particularly encouraging in the remarkable agreement exhibited between the two methods. Of the six pesticides sampled, 2 showed exact agreement, 2 showed predictions within a range of $\pm 2\%$, and the remaining 2 showed predictions within a range of $\pm 6\%$ when comparing the sedimentation to the cyclone results. The data from the six pesticides clearly supports the sedimentation method's ability to predict the respirable fraction of a bulk dust.

The data for the other 7 dusts shows high correlation ($\pm 5\%$) for 3 dusts, moderate correlation for 3 dusts ($\pm 18\%$), and low correlation for one dust ($\pm 28\%$). The decreased levels of correlation ($\pm 18\%$) for diatomaceous compounds #1, #3, and #4 were expected as these dusts carried high electrical charges when suspended in the chamber. The presence of these charges is a probable explanation for the experimental variability noted. Poor correlation for the flint dust was also anticipated as the flint dust generated a fine dust cloud during handling when using the sedimentation method,

Table VI.
Cyclone, Sedimentation and Manufacturers' Data Summary

Dust	<u>Respirable Fraction</u>		
	Cyclone	Sedimentation	Specs.
Arizona Road Dust	*	12%	13%
Flint	39%	13%	*
Clay	29%	32%	*
Micronized Amorphous Silica	29%	32%	35% **
Diatomaceous Compd. #1	47%	65%	43% ***
Diatomaceous Compd. #2	18%	20%	9% ***
Diatomaceous Compd. #3	23%	5%	1% ***
Diatomaceous Compd. #4	18%	5%	5% ***
Talc	*	62%	35% **
Pesticide Compd. #1	12%	12%	*
Pesticide Compd. #2	6%	6%	*
Pesticide Compd. #3	15%	17%	*
Pesticide Compd. #4	21%	22%	*
Pesticide Compd. #5	18%	12%	*
Pesticide Compd. #6	13%	9%	*

* Data not available

** Sampling Method Unknown

*** Sampling Method = Coulter Counter

thereby eliminating the finer respirable particles from the sampling process.

The manufacturer's respirable fraction value represents a calculated estimate based on numerical particle size distributions provided by the company. The method employed for sizing the Manville dusts was by use of a Coulter Counter. L & N Microtrac Analyzer was used to size the Arizona Road Dust. The method used by the manufacturers for analyzing the micronized amorphous silica dust and the talc dust is unknown. For 5 of the 7 dusts, the manufacturer's and sedimentation respirable data show exceptional correlation of $\pm 0-8\%$. Although variations in sampling methods reduce the strength of the comparison, studies have confirmed good correlation between the Coulter Counter method of sizing particles and that of the Andreasen pipette (19). The degree of correlation between the sedimentation technique and the L & N Microtrac Analyzer is unknown.

DISCUSSION

Sedimentation Results

The data from this study show that the respirable fraction of the test dusts can be estimated when using the Andreasen sedimentation technique. The reproducibility of the method was found to be $\pm 1\%$. The accuracy of the sedimentation method's respirable predictions was found to be $\pm 6\%$ when using the cyclone method to validate the results. This level of accuracy is dependent on adequate levels of dispersion within the sedimentation suspension. It does not apply to highly charged dusts which agglomerated in the cyclone chamber and subsequently biased cyclone results.

The data also demonstrate that the single most important factor in assuring precision and accuracy of the test results is the attainment of complete dispersion of the test dust within the sampling medium. Criteria for good dispersion include: 1) the choice of the proper sampling fluid, 2) the addition, when necessary of an appropriate dispersant, 3) proper dust concentrations within the suspension, and 4) thermal equilibrium between the sampling suspension and the laboratory environment. Application of the method is limited to particles having diameters less than 50 microns.

Cyclone Results

Each respirable fraction run was carried out using 3 two-stage respirable dust samplers and one open face sampler. Reproducibility of respirable fraction measurements was highly variable within individual runs and also from one run to the next. Some runs showed remarkable agreement of $\pm 2.0\%$ between the three cyclones' respirable fraction measurements, while others varied by as much as $\pm 32\%$. Variability in respirable fraction measurements from one run to the next for a particular dust was similar to that noted within runs, with the variation ranging from $\pm 1.9\%$ to $\pm 33\%$.

Efforts to determine the source of variability were complicated by an inability to isolate variations in cyclone performance from variations within the dust cloud in the chamber. Greater consistency was observed by using the average of the three measurements within a single run as the standard of comparison when comparing one run to the next, as well as when comparing the cyclone respirable fraction measurements to those predicted by the sedimentation method. This trend seems to indicate that the source of variability was actually within the cloud of dust generated within the chamber.

Procedures most effective in improving consistency of results included 1) Precoating the chamber with the test dust before sampling commenced, 2) eliminating the static electrical charge on the filters prior to sampling, and 3)

preventing dust overloading and breakthrough by strict monitoring of the dust concentration within the chamber and actual sampling times.

Comparison of Sedimentation Data and Manufacturers' Data

Sedimentation respirable fraction measurements were also found to be in close agreement of $\pm 8\%$ with those provided by the manufacturer. The strength of this comparison was somewhat weakened by two factors: 1) the manufacturer employed a sampling method other than that of sedimentation for sizing the particles, and 2) limited data provided by the manufacturer required significant extrapolation for some test dusts.

Comparison of the Sedimentation and Cyclone Method

Understanding why the sedimentation data can be used as an alternate to cyclone data involves many complexities which arise from the lack of association between the theory that each apparatus is based upon. The sedimentation method's operating principles are quite simple and are easily explained by the theory of Stokes' Law. In contrast, the cyclone's design is based on the human lung retention characteristics for airborne particles. The exact mechanisms of the cyclone for removing particulates are still not fully understood due to the complex design and flow patterns within the cyclone body.

Although this lack of correlation seems to exist between operating principles of the sedimentation pipette and the cyclone, there is clearly some underlying relationship between the two methods as indicated by the overwhelmingly consistent agreement between the two methods' respirable measurements of the same test dust. Although the sampling apparatuses perform in different manners, they are both capable of isolating and quantifying the same unique component of a dust, that is the respirable fraction of the total bulk material. It appears that the two methods relationship derives more from the perspective of what they sample, i.e. specific particles with specific properties, rather than the mechanics of how they sample. Therefore efforts to explain why the sedimentation data so aptly mimics that of the cyclone focus on more the particles being sampled rather than the mechanics of the apparatus.

Both the cyclone and the sedimentation method sample approximately the same distribution of particle sizes for a given dust. Each method targets fine particles having diameters < about 15 microns and effectively eliminates the coarser grained particles. When applying the method proposed by this study, the Andreasen sedimentation pipette predicts the particle size distribution for particles within the range of roughly 2-20 microns. Assuming a one hour sampling interval at a height of 5 cm from the liquid surface, the method predicts the size distribution for a siliceous dust

(density of silica = 2.65 gms/cm^3) for particles within the size range of 2.0 to 84.0 microns. However, most of the particle distribution sampled lies at the lower end of the range since the larger particles rapidly settle out of the suspension within a few seconds. After a two minute lapse, the particle sizes remaining in the suspension as predicted by Stokes' Law are those with diameters of 20.0 microns and less. This same tendency for sampling particle sizes occurs with the cyclone analysis. Upon injecting the dust into the chamber, most coarse particles immediately settle to the chamber bottom and are removed from the sampling process. This is confirmed by the rapid buildup of a thin layer of dust on the chamber bottom.

Both methods provide particle size distributions by mass by fractionating the material into different size components. In the case of the cyclone, the distribution is somewhat crude in that it is characterized by only two fractions, a respirable and a nonrespirable one, and therefore does not provide enough data points for distribution plots. On the other hand, the sedimentation method can be used to separate the dust into many size fractions, and thus provides a sufficient number of points for plotting a size distribution. For the purposes of immediate comparison to the cyclone method, the sedimentation data can be collapsed into two fractions, being the respirable and nonrespirable ones.

Both the cyclone and sedimentation provide measurements which are based upon how the particles settle within a medium, i.e. their terminal settling velocities, rather than how they would appear under a microscope. Thus the two methods measure particle sizes, whether directly or indirectly, in terms of aerodynamic diameter.

A final manner in which the two methods parallel each other is they both collect one sample which represents the total dust, and all other samples collected are quantified in terms of their ratio to the total dust sample. For the cyclone, the total dust sample is that collected on the open face filter, whereas, with the sedimentation pipette, the first sample drawn represents the total dust sample.

CONCLUSIONS

The data from this study show that it is not mandatory to disperse a sample of the bulk powder in air in order to estimate the respirable fraction. This is of particular significance in the field of industrial hygiene since methods to generate a uniform dust into a test chamber when using the cyclone method are lacking. Furthermore, the sedimentation method fails to show the high degree of variability in respirable fraction measurements that so commonly is associated with the cyclone method.

Additional advantages of the sedimentation method are that it is simple to perform and does not require highly skilled personnel for carrying out the analyses. The method is quick, reproducible and accurate. The apparatus is relatively inexpensive and does not require calibration or routine maintenance. The method also minimizes the inhalation exposures to the user that routinely occur with the cyclone method since the analysis is carried out in a liquid medium rather than air.

The application of the sedimentation method for making respirable fraction determinations of bulk siliceous materials is extensive. Commercial products containing silica are abundant and include cosmetics, toiletries, powdered drugs, paints, cements, and food powders. Manufacturers of these products can use the method to determine that component of the dust that represents an

inhalation hazard to exposed individuals. Subsequently, the fraction separated during sedimentation analysis can be analyzed by x-ray diffraction for crystalline silica to determine the respirable fraction of that particular component. These results could then be used to assess whether the product requires labeling as a carcinogen under the federal carcinogenic labeling requirements. In some cases, early detection of high respirable fractions of crystalline silica within a product can provide manufacturers an opportunity to reformulate their product so as to avoid the adverse commercial impact caused by a carcinogenic warning label.

RECOMMENDATIONS FOR FURTHER RESEARCH

The dusts analyzed by this study include flint, clay, diatomaceous earths, talc, clay, Arizona road dust, several pesticides and a micronized amorphous silica sample. Particle shapes comprising these dusts include plates, spheres, rods, and many with geometries showing varying magnitudes of deviation from perfect spheres. Further research in the application of the sedimentation method to other materials would be of significance, particularly for dusts characterized by fibers.

It would also be of interest to investigate the feasibility of using the sedimentation method in place of the cyclone method for hazards evaluation of industrial inhalation exposures as well as industrial compliance with federal and state respirable dust standards. For example, high volume samples of siliceous dusts could be collected, and then analyzed for the respirable fraction using the Andreasen sedimentation method.

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APPENDIX A

Equipment List for Sedimentation Analysis:

Andreasen sedimentation pipette
Drying oven
Balance
Ventilation hood
Aluminum evaporation pans
Thermometer
Stopwatch
Microscope
Microscope slides
Dispersing agents (Sudless detergent, ethyl alcohol, etc.)

Sedimentation Procedure

1. Using Stokes' equation, determine the sampling times and depth necessary for collecting the particle size range of interest.
 2. Record the date, temperature, and air pressure. The medium must be with in thermal equilbirum with the air.
 3. Adjust the drying oven to 95°F. If the temperature of the oven is too high, the samples will boil and sampling material may be lost.
 4. Number and label the aluminum evaporation dishes.
 5. Zero the balance. Weigh each pan and record the weight to the nearest milligram.
 6. Conduct dispersion analyses as previously described.
 7. Collect and weigh the desired amount of sample from the bulk material. The concentration of the test material should be 0.75-2% by volume when diluted in the sedimentation vessel filled to the 20 cm or 550 ml graduation mark.
 8. Adjust the pipette to the desired sampling height.
 9. Add the liquid medium to the cylinder to approximately the 15 cm mark.
 10. Pour the sample into the cylinder through a funnel to prevent loss of test material.
 11. Adjust the suspension height with additional liquid until it reaches the 20 cm graduation mark when the pipette is inserted in the flask.
 12. Cover the vent hole in the stopper and agitate the suspension for two minutes.
 13. Start timing the experiment and immediately draw the first sample to the 10 ml mark on the pipette by applying pressure to the rubber bulb at a slow and steady rate. Twenty seconds is a reasonable sampling time.
- Note: Every effort should be made to ensure the accuracy and precision of this first sample as the first sample serves as the basis upon which all sample calculations are made.
14. Drain the sample into the aluminum evaporation dish and place in drying oven.

15. Continue to draw samples at the predetermined sampling times. All samples should be collected in the same manner, that is, over a twenty second interval, and to the 10 ml mark on the pipette.
16. After the samples have dried, remove from oven and allow to cool.
17. Zero the balance. Weigh each aluminum pan with sample and record the weight to the nearest milligram.

Calculations

1. Subtract the original weight of the aluminum pans from the weight of the pans containing the dried sample to determine the amount of sample collected in each pan.
2. For each sample, determine the percentage by weight of the original sample. The weight of the first sample represents the original sample weight.
3. Using equation (6) on page 16, determine the particle diameter that represents the largest particle present in the sample by plugging in the appropriate values for each variable.
4. Plot the results on log-probability paper with grain size (D) on the y-axis and the cumulative percentage by weight on the x-axis. The percentage by weight of any grain size can be determined by referring to the log-probability curve.

Equipment List for Cyclone Respirable Fraction Analysis:

10-mm nylon cyclones
Sampling chamber
Rotameters
Sampling pump
Flexible clear tubing
Clamps for tubing
Cyclone assemblies
Ring stands
Mixing fan
Type AA 37 mm PVC membrane filters
Cellulose back-up pads for filters
Two and three-piece lucite filter cassettes
Static eliminator
Balance
Aspirator
Pressure source
Bone-tip tweezers
Beaker
Stopwatch
Respirator
Ventilation hood

Cyclone Procedure

1. Calibrate pump and rotameters against primary standard.
 2. Thoroughly clean chamber and all sampling equipment.
 3. Weigh out approximately 15-20 grams of the sample material in a glass beaker.
 4. Seal the chamber.
 5. Aspirate the 15-20 gms of sample material into the chamber, allow the fan to run for about 10 minutes, and then allow particles to settle. Coating the chamber before sampling is a critical step in obtaining accurate and consist sampling results.
 6. Zero the balance.
 7. Pass filter over static eliminator and weigh filter to the nearest microgram. Record the filter weight as the average of five successive readings.
- Note: Do not touch filters with fingertips. Bone-tipped tweezers are recommended for filter handling.
8. After weighing, immediately load each filter into the lucite cassettes. Each filter should be supported by a cellulose back-up pad to prevent breakthrough while sampling. Load filters for respirable sampling into two-piece cassettes, and filters for open face sampling into three-piece cassettes. Seal the cassette with tape and label for identification. It is common practice to label runs with a letter of the alphabet followed by successive numbers. For example, the cassettes within one run would be labeled A001, A002, A003, etc.
 9. Attach the loaded cassettes to cyclone assembly.
 10. Connect tubing from sampling pump to open-face and respirable sampling cassettes.
 11. Place rotameters in line to indicate actual pump flow-rates.
 12. Place samplers within brackets in the chamber. Respirable samplers should be in an upright position. Open face samples should be oriented with the filter located vertically. Record the distance from the chamber bottom to the filters. All filters should be located at the same distance above the chamber bottom.

13. Transfer a sample from the bulk material to the glass beaker and weigh on balance.

14. Aspirate the sample into the chamber and allow the mixing fan to run for one minute.

15. Turn off the fan, note the time, and commence sampling. Sampling time will vary with the concentration of the sample within the chamber and inherent properties of the material itself. Filter loading should not exceed two milligrams. Visual inspections of the open-face filter while sampling will help assure that dust overloading on the filters does not occur.

16. When the sampling time has elapsed, turn off the pump, and remove the cassettes from the chamber. Do not invert the sampler assembly at any time. Inversion of the cyclone may deposit over-sized material collected within the cyclone body onto the respirable filter. Return the top to the open-faced filter, and cap both sides of all cassettes.

17. Zero the balance, and weigh the filters with the collected samples. Record the weights to the nearest microgram.

18. Return the filters to the cassettes, and prepare for shipment to laboratory if free silica analysis is required. Cap and label all cassettes.

Cyclone Respirable Fraction Calculations

1. Divide the sample mass collected on the respirable sample filters by the sample mass collected on the open face filter to determine the respirable fraction of the bulk material when airborne.

APPENDIX B

Occupational Safety & Health Administration
Permissible Exposure Limits for Crystalline Silica (Quartz)
(33)

Substance	Mg/M ³ *
Quartz (respirable)	$\frac{10 \text{ mg/m}^3}{\% \text{SiO}_2 + 2}$
Quartz (total dust)	$\frac{30 \text{ mg/m}^3}{\% \text{SiO}_2 + 2}$

Cristobalite (respirable): Use 1/2 the value calculated from the formula for quartz.

Tridymite (respirable): Use 1/2 the value calculated from the formula for quartz.

Example: A respirable dust sample contains 3.5% SiO₂ (quartz). The PEL would be calculated as follows:

$$\frac{10 \text{ mg/m}^3}{3.5 + 2} = 1.82 \text{ mg/m}^3$$

For this particular dust, airborne levels of respirable quartz dust in excess of 1.82 mg/m³ would violate the OSHA PEL for respirable crystalline silica.

* Milligrams/meter³

**American Conference of Governmental Industrial Hygienists
Threshold Limit Values for Silica (24)**

Substance	Mppcf*	TWA (Mg/M ³)**
Crystalline Silica:		
Quartz (respirable)	$\frac{250}{\%SiO_2 + 5}$	0.1 mg/m ³
Cristobalite (respirable)		0.05 mg/m ³
Tridymite (respirable)		0.05 mg/m ³
Amorphous Silica:		
Diatomaceous earth (uncalcined, total dust)		10 mg/m ³

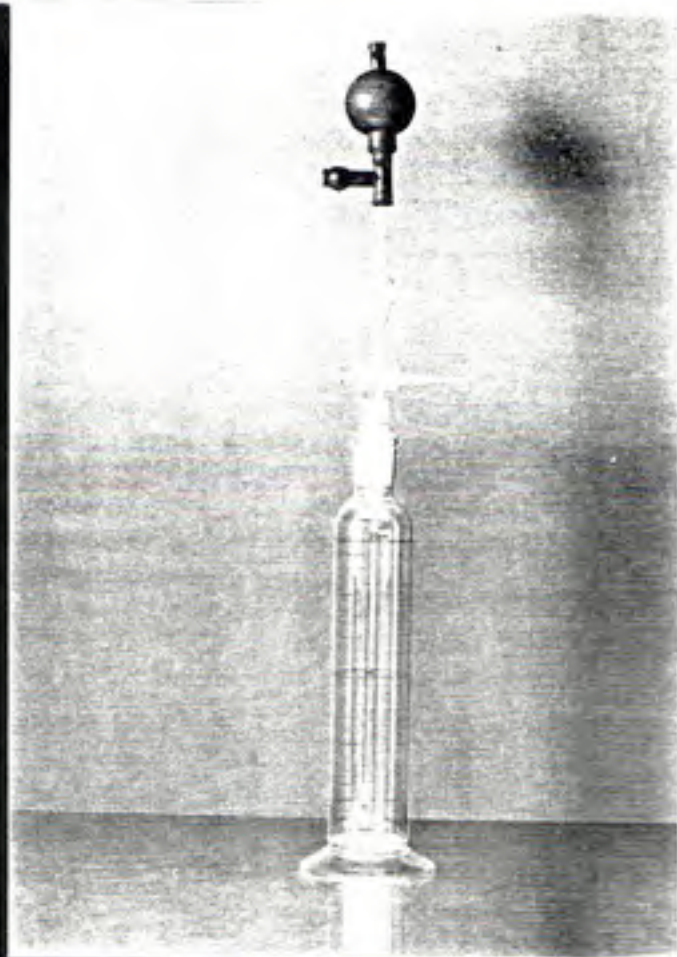
Note: An employee's exposure to any of the above substances in any 8-hour work shift of a 40-hour work week shall not exceed the 8-hour time weighted average limit.

* Million particles per cubic foot

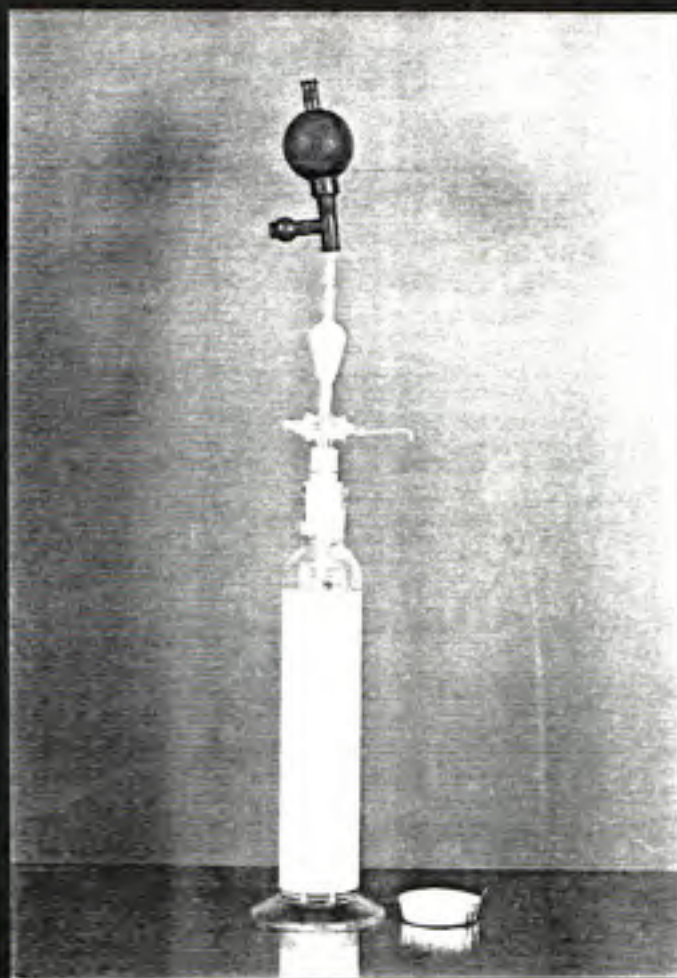
** Time weighted average in milligrams/meter³

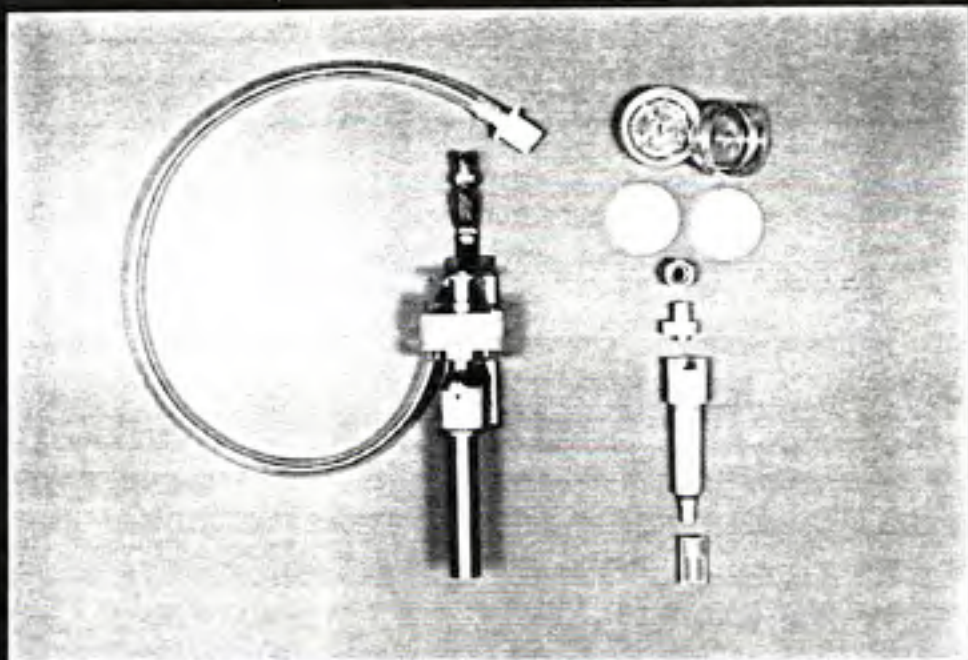
APPENDIX C

Photograph A.
Andreasen Sedimentation Pipette.

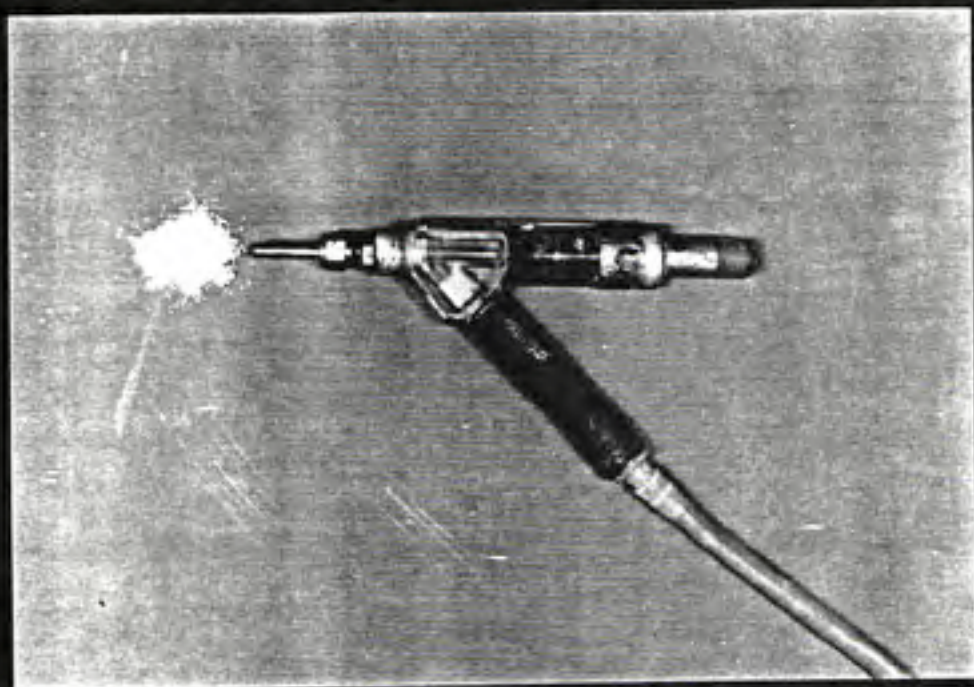


Photograph B.
Andreasen Sedimentation Pipette
with Suspension.

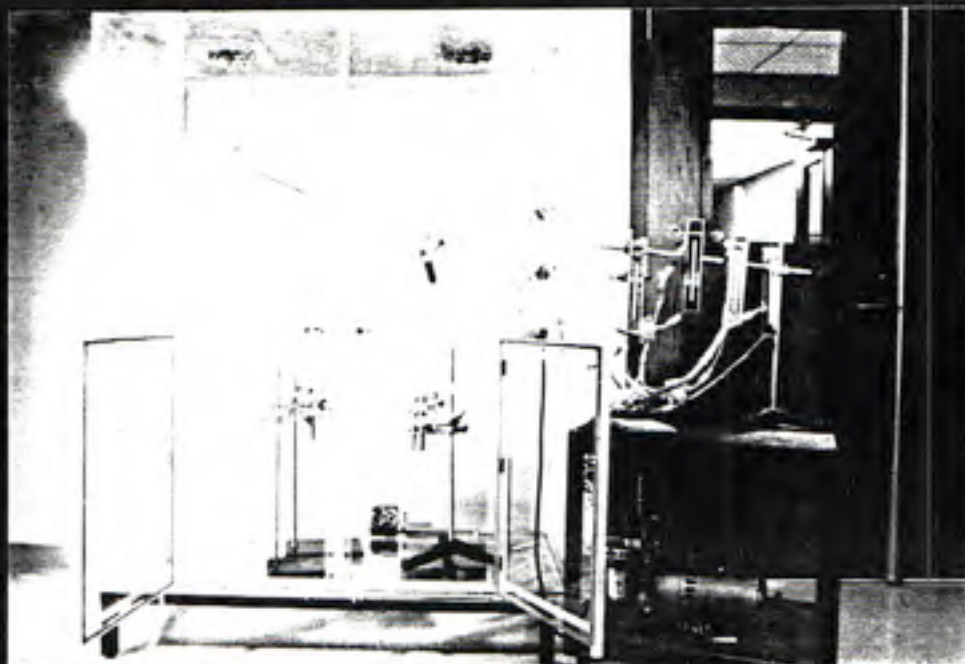




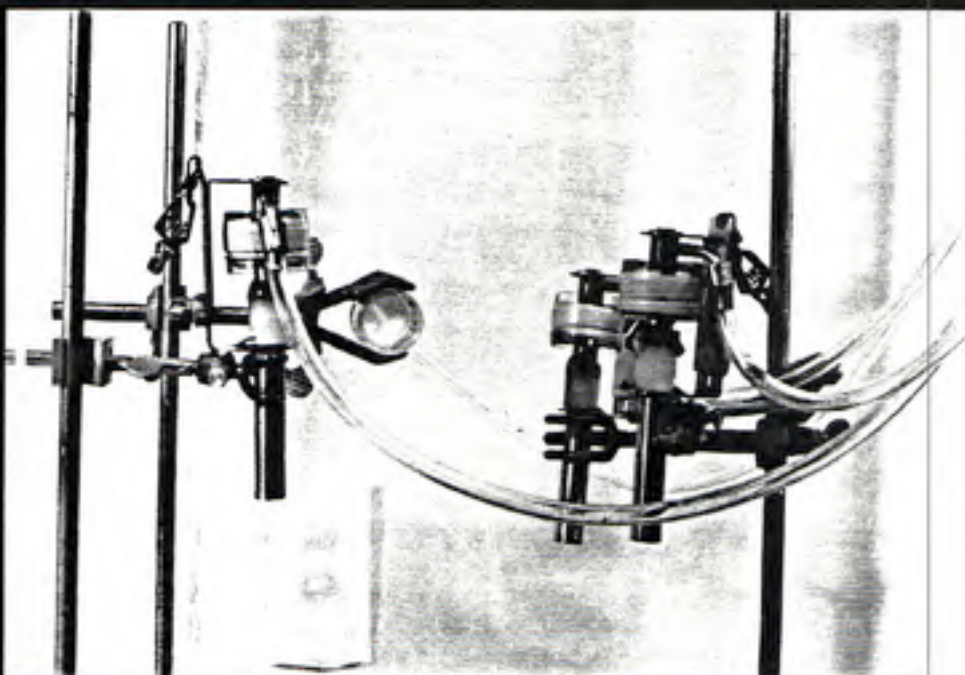
Photograph C.
10-mm Nylon Cyclone and Assembly.



Photograph D.
Aspirator Device



Photograph E.
Experimental Chamber and Cyclone
Set-up.



Photograph F.
Experimental Cyclone Set-up.