Acknowledgments

I would like to express my grateful thanks to Dr Brian H. Kaye for introducing me to the fascinating study of particle size analysis. My thanks are also due to numerous workers in this field for the helpful discussions we have had. Bradford University has provided me with a well-equipped laboratory in which, in teaching others, I have learnt some of the secrets of this science. One of my students was Mr T.S. Krishnamoorthy and the chapter on gas adsorption is taken from his M.Sc. thesis. At Bradford, Mr. John C. Williams has always had the time to offer helpful advice and criticism. I make no apology for taking up so much of his time since his advice was invariably good and whatever virtue this book possesses is due, in part, to him.

My thanks are also due to holders of copyright for permission to publish and to many manufacturers who have given me full details of their products.

Finally, I would like to thank my wife for her forbearance while the writing of this book has been in progress.

Terence Allen

1 Sampling of powders

1.1 Introduction

There are many instances where estimates of population characteristics have to be made from an examination of a small fraction of that population and these instances are by no means confined to the field of powder technology. Regrettably, there are many powder technologists who still assume that sample selection procedure is unimportant. This results in the analyst being frequently presented with hastily taken, biased samples on which he devotes a great deal of attention to derive precise results which do not reflect the characteristics of the bulk powder. It is essential that the samples selected for measurement should be representative of the bulk in grain size distribution and the relative fractions of their various constituents, irrespective of whether a physical or chemical assay is to be carried out, since these characteristics are frequently inter-dependent. The magnitude of the problem may be realized when one considers that the characteristics of many tons of material are assessed on the basis of analyses carried out on grams or even milligrams.

The probability of obtaining a sample which perfectly represents the parent distribution is remote. If several samples are taken their characteristics will vary and if these samples are representative, the expected variation may be estimated from statistical analysis. However, the sampling equipment will introduce a further variation which may be taken as a measure of sampler efficiency. Imposed on this there may also be an operator bias. The reduction from bulk to measurement sample may be conveniently divided into the four stages illustrated below.

<table>
<thead>
<tr>
<th>Process or delivery of materials</th>
<th>Gross sample</th>
<th>Laboratory sample</th>
<th>Measurement sample</th>
</tr>
</thead>
<tbody>
<tr>
<td>$(10^n)$kg</td>
<td>(kg)</td>
<td>(g)</td>
<td>(mg)</td>
</tr>
</tbody>
</table>

Bias at any of the reduction stages will adversely affect the final analysis.

1.2 Theory

The ultimate that may be obtained by representative sampling may be called the perfect sample; the difference in population between this sample and the bulk may be
2 Particle Size Measurement

A powder to be sampled may be considered as made up of components A and B. The probability that the number fraction \( p \) of the bulk in terms of A shall be represented by the corresponding composition \( p_i \) of a perfect sample can be computed from the number of particles of A and B in the sample \( n \) and in the bulk \( N \):

\[
\text{Var}(p_i) = \frac{p(1 - p)}{n} \left(1 - \frac{n}{N}\right)
\]

(1.1)

The theoretical standard deviation \( \sigma_1 \) is equal to the square root of the variance. Assuming a normal distribution of variance, if representative samples are taken from the bulk, 68.3% of the samples should not vary from the true number fraction of A by more than one standard deviation and 95.4% should lie within two standard deviations of the mean. These percentage values are taken from a table of the area under a normal curve. These tables may be found in any statistics book [29, 30].

These areas give the probability of an event occurring and are derived from the integration of the normal probability equation:

\[
d\Phi = \frac{dx}{\sqrt{2\pi}\sigma_1}\exp\left(-\frac{x^2}{2\sigma_1^2}\right)
\]

(1.2)

where \( x = (p - \bar{p}) \) and \( \bar{p} = \text{the 50% probability level (median)} \).

The area between \( p = \bar{p} \) and \( p = \bar{p} + \sigma_1 \) equals 0.3413, hence 68.26% of the samples should deviate from the mean by less than one standard deviation.

Instead of the number fractions it is more convenient to assess sample and bulk compositions in terms of weight fractions \( P \) and \( P_i \) giving [2]:

\[
\text{Var}(P_i) = \frac{P(1 - P)}{w} \left[P w_B + (1 - P) w_A\right] \left(1 - \frac{w}{W}\right)
\]

(1.3)

where \( W \) and \( w \) are the bulk and sample weight respectively and \( w_A \) and \( w_B \) are the weights of individual grains of components A and B.

Example

Consider a binary powder made up of equal weights of particles of weight 0.05 and 0.10 g. Determine the expected variation, assuming perfect sampling, for a 50 g sample removed from a bulk of 800 g.

From equation (1.3):

\[
\text{Var}(P_i) = \frac{0.50 \times 0.50}{50} \left[0.50(0.10) + 0.50(0.05)\right] \left(1 - \frac{1}{16}\right)
\]

\[
= 3.52 \times 10^{-4}
\]

\( \sigma_1 = 1.88\% \)

From equation (1.1):

\[
\left(\text{derived data; } p = \frac{1}{3}, n = 750\right)
\]

\[
\text{Var}(P_i) = \frac{1}{750} \left(\frac{1}{3} \times \frac{2}{3}\right) \left(1 - \frac{1}{16}\right)
\]

\[
= 2.78 \times 10^{-4}
\]

\( \sigma_1 = 1.67\% \)

Assuming a normal distribution of variance, the probability of representative samples lying within one standard deviation of the mean is 68.3%, and 95.4% should lie within two standard deviations of the mean. Three results in a thousand will fall outside three standard deviations.

On a weight basis, of the 50 g withdrawn, the weights of each component at the three probability levels above, are (in grams): 25 ± 0.94, 25 ± 1.88, 25 ± 2.82.

On a number basis, of the 750 particles withdrawn, the numbers of small particles at the above three probability levels are: 500 ± 12.5, 500 ± 25, 500 ± 37.5.

On a percentage basis by number, \( p \pm \sigma_1 = (66.7 \pm 1.67)\% \) of 750.

These equations may be used as a basis from which to assess the efficiency of a real, non-ideal sampling device. In this case the variance of the sample assay, \( \text{Var}(P_n) \), will be greater than \( \text{Var}(P_i) \) due to the non-ideality unless the bulk powder is homogeneous.

\[
C = \frac{\text{Var}(P_i)}{\text{Var}(P_n)}
\]

(1.4)

This should approximate to unity when sampling errors are low, hence the sampling efficiency may be defined as 100C. Sample variance \( \text{Var}(P_n) \) can be calculated from experimental results:

\[
\sigma_n = \sqrt{\frac{(x_i - \bar{x})^2}{n}}
\]

(1.5)

where \( \sigma_n \), the sample standard deviation, equals the square root of the sample variance. \( \bar{x} \) is the true percentage of A or B particles by weight in the mixture, \( x_i \) is the percentage in the \( i \)th sample and \( n \) is the number of samples examined. If \( \bar{x} \) is unknown, it may be approximated to, using the equation:

\[
\bar{x} = \frac{\sum_{i=1}^{n} x_i}{n}
\]

(1.6)

In this case the denominator in equation (1.5) should be replaced by \( (n - 1) \).

Theoretical and experimental errors may be separated using the equation:

\[
\sigma^2 = \sigma_n^2 - \sigma_1^2
\]

(1.7)
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The maximum sample error ($E$) can be expressed as a percentage of the bulk assay.

$$E = \frac{\sigma}{P} \times 100$$  \hspace{1cm} (1.8)

It should be noted that, since sample variations will probably be related to the particle size distribution of the powder to be sampled, it is not possible to measure the absolute efficiency of a sampling technique. Sampling errors may be of two kinds: random fluctuations and a steady bias due to faulty design of the sampling technique.

Numerical example

Any powder can be considered as being made up of two components, the fraction above and below a certain size. For a reduction of 16 to 1 during sampling with $W_A = 0.10$ g, $W_B = 0.05$ g and $w = 50$ g, equation (1.3) becomes:

$$\text{Var} (P_i) = \frac{3}{160} W_B \left( P^3 - 3p^2 + 2P \right).$$

The maximum value of the variance, derived by differentiation and equating the differential to zero, occurs for $P = (1 - 1/\sqrt{3})$ giving $\text{Var} (P_i) = 0.000361$ and $\sigma = 1.90\%$.

This means that if 50 g samples are repeatedly removed from a bulk powder of 800 g and replaced without loss (with $W_A = 0.10$ g, $W_B = 0.05$ g), the maximum standard deviation at any percentage level would be equal to or less than 1.9%, provided no sampling bias existed.

This deviation is similar to what one would expect if one took samples of balls from a well-mixed bag containing one-third red balls and two-thirds white, but otherwise identical. If 18 balls were removed ($n/N$ very small), from equation (1.1), $q_1 = 11.1\%$ or, $S = \sqrt{np(1-p)} = \sqrt{(18 \times \frac{1}{3} \times \frac{2}{3})} = 2$ balls. That is, the minimum number of white balls one would expect would be six, i.e. the mean minus three standard deviations.

The probability of having $r$ white balls is given by:

$$\binom{20}{r} \left( 1 - \frac{1}{3} \right)^r \left( \frac{1}{3} \right)^{18-r}$$

If $r = 7$, $P(7, 18, \frac{2}{3}) = \binom{18}{7} \left( \frac{1}{3} \right)^7 \left( \frac{2}{3} \right)^{11} = 1.05\%$

Further details on statistical analysis can be found in standard texts [29, 30].

If 1800 balls were removed the standard deviation would fall to 1.11\% (i.e., 20 balls).

If the experimental deviation exceeded this, one would suspect either a non-random mix or selective sampling.

It is obvious that the larger the sample, the smaller will be the deviations. The size of a practicable laboratory sample is usually minute in relation to the whole of the material being examined. Thus the laboratory sample itself is subject to a degree of variation which is unrealistically large. There are two ways of reducing this variation;

1.3 Golden rules of sampling

There are many possible situations in which a sample has to be obtained and conditions often necessitate the use of inferior techniques. Some principles can however be laid down, and they should be adhered to whenever possible:

- **Rule 1.** A powder should be sampled when in motion.
- **Rule 2.** The whole of the stream of powder should be taken for many short increments of time in preference to part of the stream being taken for the whole of the time.

Observance of these rules coupled with an understanding of the manner in which segregation may have occurred during the previous treatment of the powder will lead to the best sampling procedure. Any sampling method which does not follow these rules should be regarded as a second-best method liable to lead to errors.

1.4 Bulk sampling

There are a very large number of possible systems from which the gross sample has to be abstracted, so it is impossible to lay down instructions which will meet all situations. The problem may be to obtain a sample from continuous streams, batches, packets, heaps or trucks. The difficulty is that when a particulate material is handled, segregation may take place. The most important segregation-causing property is particle size and the problem is enhanced with free-flowing material. When poured into a heap, the fines tend to collect at the centre of the heap. In vibrating containers a layer of coarse material tends to collect near the surface; even if a large particle is more dense than the smaller particles in which it is immersed, it can be made to rise towards the surface. This can be demonstrated by placing a one-inch diameter steel ball in a beaker which is then filled with sand to a depth of about two inches. By stroking the base of the beaker gently with one hand, the steel ball can be made to rise to the surface of the sand. Since the surface region is always rich in coarse particles, samples should never be removed from the surface region. An understanding of these tendencies to segregation prevents careless practice in obtaining samples.

When sampling is undertaken from a continuous stream, the sampling may be continuous or intermittent. In continuous sampling a portion of the flowing stream is split off and frequently further subdivided subsequently. In intermittent sampling the whole stream is taken for small increments of time at fixed time intervals.

These increments are usually compounded and samples for analysis taken from this gross sample. Consignment sampling is carried out on a single consignment (e.g. a truck-load or wagon-load).
A general rule in all sampling is that whenever possible the sample should be taken when the powder is in motion. This is usually easy with continuous processes; with consignment sampling it may be possible during the filling or emptying of storage containers.

1.4.1 Sampling from a moving stream of powder

In collecting from a moving stream, care should be taken to offset the effects of segregation. For example, the powder may be sampled as it falls from the end of a conveyor; this is one of the best methods of sampling and should be adopted whenever possible. The powder on the conveyor will probably show two forms of segregation. If the powder was charged on to the conveyor belt from a centrally placed feeder or hopper outlet, the fines will tend to concentrate at the centre of the belt and the coarse particles will roll to the outer edges. If there has been any vibration of the belt, larger particles will tend to rise to the top of the bed of powder.

Each increment should be obtained by collecting the whole of the stream for a short time. Care must be taken in putting the sampler in and out of the stream. Figure 1.1 shows correct and incorrect methods of doing this.

Unless the time, during which the sample receiver is stationary in its receiving position, is long compared with the time taken to insert and withdraw the sampler, the method shown in figure 1.1(a) will lead to an excess of coarse particles as the surface region of the stream, usually rich in coarse particles, is sampled for a longer time than the rest of the stream. The method shown in figure 1.1(b) is not subject to this objection. If the method shown in the figure (b) is not possible due to some obstruction, the ratio of stationary time to moving time for the receiver should be made as large as possible.

In many cases it is not possible to collect the whole of the stream as this would give too large an amount to be handled. The best procedure is to pass through the stream a sample collector of the form shown in figure 1.1(c).

The width of the receiver, b, will be chosen to give an acceptable weight of sample but must not be made so small that the biggest particles have any difficulty

---

Fig. 1.1 Sampling from moving streams. (a) Bad sampling technique (b) Good sampling technique (c) Sampling procedure to be adopted for mass flow rate.

Fig. 1.2 Full-stream (GECO) sampler.
in entering the receiver. Particles that strike the edges of the receiver are likely to bounce out and not be collected so that the effective width is \((b - d)\), where \(d\) is the diameter of the particles. The effective width is therefore greater for small particles than for large particles. To reduce this error to a reasonable level, the ratio of box width to the diameter of the largest particle should be made as large as possible with a minimum value of 20:1. The depth \(d\) of the receiver must be great enough to ensure that it is never full of powder. If the receiver fills up before it finishes its traverse through the powder, a wedge-shaped heap will form a size-selective particle collector. As more material falls on the top of the heap the fine particles will percolate through the surface of the heap and be retained, whereas coarse particles will roll down the sloping surface and be lost. The length of the receiver \(c\) should be sufficient to ensure the full depth of the stream is collected.

A proprietary example of this type of sampler is the GECO manufactured by John Smith & Co. Ltd, Horsham, Sussex, diagrams of which are shown in figures 1.2 and 1.3.

This equipment is satisfactory for many applications but it has limitations which restrict its use. These are:

1. Although comparatively readily designed into a new plant, it is frequently difficult and expensive to add to an existing plant.

(2) The quantity of sample obtained is given by the product of slit width and plant rate divided by the cutter speed and is independent of stream shape or area. This proportionality to the plant rate can be inconvenient when the plant rate is subject to wide variations and a fixed sample is required. On the other hand, where a plant daily average is required, this is a necessary requirement of the sampler. Also the quantity of sample may be inconveniently large.

3. It is difficult to enclose the sampler to the extent required to prevent the escape of dust and fume when handling a dusty product.

Figure 1.4 shows a sampler designed by ICI [1] to sample a dusty material, sampling taking place only on the return stroke. This is suitable provided the trough does not overfill. For this reason the constant volume sampler, the action of which can be readily understood from figure 1.5 cannot be recommended. The slide valve sampler (figure 1.6), developed by the same company, also has defects which render it unsuitable for collecting size-representative samples.
Fig. 1.5 Constant volume sampler.

Fig. 1.6 Slide valve sampler.

Fig. 1.7 Automatic sampling from a hopper.
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A variant of this problem is encountered in sampling from preweighed batches; such an automatic sampling system is shown in figure 1.7. From a consideration of this diagram, it can be seen that the device is liable to be highly size-selective.

In a sampling device described by Clarke [21] the slide valve is replaced by an Archimedean screw which samples continuously. A variation of this, the Simon Limpet Autosampler, is designed for continuous collection of a representative sample of freely flowing material such as wheat, maize, other seeds and their flows or other products.

1.4.2 Sampling from a conveyor belt or chute

When a sample is to be collected from a conveyor belt, the best position for collecting the increments is where the material falls in a stream from the end of the belt. If access at such a point is not possible, the sample must be collected from the belt. The whole of the powder on a short length of the belt must be collected, and again it must be borne in mind that the particles at the edge of the belt may not be the same size as those at the centre, and particles at the top of the belt may not be the same as those at the bottom. If the belt can be stopped, the sample can be collected by inserting into the stream a frame consisting of two parallel plates shaped to fit the belt; the whole of the material between the belts is then swept out. With the belt in motion, the same procedure could be adopted but it is difficult to do this by hand. Some mechanical samplers are available for collecting a sample from a moving conveyor belt (figure 1.8); before using such a device its action should be carefully examined to ensure that it collects the whole of the stream and does not, for example, leave a thin layer on the belt.

![Diagram](image)

**Fig. 1.8 Automatic sampler for belt conveyor.**

1.4.3 Sampling from a bucket conveyor

In sampling from a bucket conveyor no attempt should be made to collect part of the material from a bucket. Each increment should consist of the whole of the contents of a bucket. If this is too large an amount for convenient handling, it should be reduced in size by one of the methods described later.

1.4.4 Bag sampling

Suppose an analysis is required from several tons of material which is available in sacks. Should a sack be selected at random with the hope that it is representative of the bulk? Having selected a sack how should a sample be withdrawn for subsequent measurement?

In an analysis of this problem Kaye [4] suggests that several sacks should be selected in order to obtain a representative sample. These may be selected systematically, i.e. the 100th, 200th, 300th and so on or, preferably, using a table of random numbers.

The sacks may be examined individually in order to determine whether the variation between sacks is of an acceptable level or combined in order to determine an average. In either case it is necessary to obtain representative samples from each sack. For this purpose Kaye recommends a thief sampler but warns that this may easily give a biased sample.

The best methods are undoubtedly those that conform with the 'golden rules of sampling' such as the LADAL Bulk Spinning Riffler and the expense of installing and using this type of equipment has to be balanced against the cost of rejecting good material or accepting poor material by using a more inexpensive technique.

1.4.5 Sampling spears

Scoop sampling is the most widely used method of sampling because of its simplicity. A presupposition is that the powder, at the point at which the scoop is inserted, is representative of the bulk. Accuracy should be increased by taking more than one sample. These should be examined separately in a preliminary investigation and combined in later investigations if the variance between the samples is at an acceptable level. This technique is improved if samples from the body of the material are included and this may be effected with the aid of a sampling spear. Three types are available (figure 1.9); in type 1 the sampling chamber runs the full length of the spear; in type 2 the sampling chamber is at the end of the spear and in type 3 there are separate sampling chambers along the length of the spear. The spear is thrust into the powder with the inside chamber closed off. When in position the inner tube is rotated to allow powder to fall into the inner chamber. When the sampling chamber is full the inner tube is turned to the closed position and the spear is withdrawn. Possible segregation throughout the bed may be investigated with type 3, an average value for the length of the spear with type 2 and a spot sample obtained with type 1.
1.4.6 Sampling from wagons and containers

It is very difficult, in fact practically impossible, to obtain a satisfactory sample from a wagon or container, because of the severe segregation that would almost certainly occur in filling the wagon and in its subsequent motion. A method for removing samples that avoids some of the worst errors is described in [5]. No increments are to be collected at less than twelve inches below the surface; this avoids the surface layer in which extreme segregation will probably have occurred due to vibration. In removing the samples there must be no surfaces down which particles can slide; this is achieved either by pushing in a sampling probe which extracts the sample (figure 1.9), or by removing particles by hand in such a way that no sliding occurs; when the sample region is exposed the sample can be extracted.

![Sampling points for a wagon or truck (sampling from bottom of granular excavation).](image)

Increments should be extracted at eight points as shown in figure 1.10. This method of obtaining a sample is mentioned here because it is possible that there may be circumstances in which there is no alternative but to use it, but this must not be taken to imply that such methods will give satisfactory sampling. Every effort should be made to avoid this method and to use one that satisfies the two 'golden rules'. If a powdered material is in a container, the container has been filled and presumably is going to be emptied. At both these times, the powder will be in motion, and a more satisfactory sampling procedure can then be used.

1.4.7 Sampling from heaps

There is only one sound piece of advice to give regarding sampling from a heap. 'Don't Never!' Examination of the cross-section of a heap of powder containing particles of different sizes shows that there is a very marked segregation, the fine particles being concentrated in a region near the axis of the heap, with the coarse particles in the outer part of the heap.

The photograph (figure 1.11) of the cross-section of a heap of granules of two sizes shows the segregation of powder when it is poured into a heap. Although segregation
does not occur on such a scale with sub-sieve granules, it indicates the care that must be taken when sampling from a heap. Sometimes a sample has to be obtained from a powder at rest, an indication that the sampling is taking place at the wrong time, since it must at some time have been in motion. In this case, the sample should be compounded from incremental samples and a lower sampling efficiency is to be expected. Figure 1.12 shows a pneumatic probe for extracting such samples.

In one case a binary mixture of equal weights of particles of two sizes, having a diameter ratio of 2.8, was poured into a heap and the concentration of fine particles in different parts of the heap was found to vary between 1 and 70%. Any attempt to find the composition of the whole heap from measurements based on sampling from a heap has, therefore, little chance of giving an accurate answer.

The solution to the problem of sampling from a heap lies in the fact that the powder must have been poured to form the heap, and this is the time when the sample should be collected; increments can be withdrawn from the falling stream by one of the recommended methods.

1.5 Slurry sampling

Clarke [21] discusses the problems of sampling from liquid streams and describes a parallel-sided scoop for extracting a sample size which is proportional to flow rate in an open channel.

Hinde and Lloyd [25] are more interested in extracting samples for continuous on-line analysis. They state that the process streams from industrial wet classifiers can vary in volume flow rate, solids concentration and particle size distribution. Any sampling technique should be capable of coping with these variations without affecting the representativeness of the extracted sample.

For batch sampling, automatic devices are available where a sampling slot is traversed intermittently across a free-falling slurry. Unfortunately it is difficult to improvise with this technique for continuous sampling since such samplers introduce pulsating flow conditions into the system.

Osborne [26] described a sampler which consists of a narrow slot continuously rotated on an axis parallel to the slurry flow (figure 1.13(a)). Cross [27] used a slotted pipe mounted vertically in the overflow compartment next to the vortex finder of a hydrocyclone (figure 1.13(b)).

Since most continuous size analysers require a small constant volume flow rate further subdivision is often necessary. Osborne's solution (figure 1.13(c)) was to feed the sample stream to a well-agitated sampling tank and withdraw a representative sample at a controlled flow rate.

Autometrics [28] analyse the whole of the extracted sample (figure 1.14(a)). Gaps are left below the weirs to prevent sanding. Sub-division of the sample stream (for calibration purposes) can be accomplished very successfully by syphoning from a vertical fast-flowing stream (figure 1.14(b)).

Preferably the whole process stream should be used for on-line analysis as discussed in Chapter 19.
Fig. 1.13 Sampling devices: (a) rotating slot of Osborne [26]; (b) slotted pipe of Cross [27]; (c) sampling tank of Osborne [26]. (From [25] with permission)

Fig. 1.14 More sampling devices: (a) Autometrics design for direct sampling of process stream; (b) Autometrics design for extracting small calibration samples [28]. (From [25] with permission)
1.6 Sample dividing

The gross sample is frequently too large to be handled easily, and before sending it to the laboratory for testing, it may have to be reduced to a more convenient weight. Obviously this must be done in such a way that the laboratory sample has the same size grading as the gross sample. The principles involved here are the same as those already discussed when considering the collection of the gross sample. The first step is to realize the difficulties involved, and to understand the ways in which the segregation of the powder is likely to occur. The sampling procedure must then be designed so as to minimize the effects of segregation. The two 'golden rules' of sampling mentioned earlier apply equally here. The best method of sample dividing is:

(1) Get the powder into motion in a stream.

(2) Each increment should be obtained by collecting the whole of the stream for a short time.

Increments are collected at equal time intervals and put together to form the laboratory sample. To obtain the best results, the stream should be made as homogeneous as possible. If complete homogeneity of the stream is achieved, then clearly every increment will have the same size grading as the whole of the material and, in such an imaginary case, it would be enough to collect one increment. If there are considerable changes in the size grading of the material from one part of the stream to another, the probability of getting increments with the same size grading as the whole is reduced. Everything must be done to make the process of sample dividing easier and, by giving thought to the handling of the powder fed to a sample divider, more accurate results can be obtained. For example, if the sample divider is fed from a hopper, this hopper should have steep sides (at least 70°) so that mass flow is likely to occur when it is emptied, and the hopper should be filled in such a way that size segregation does not occur; this can best be done by moving the pour point about so that the surface of the powder in the sample divider is always more or less horizontal and no conical heap is formed. Several sample-dividing devices have been recommended and some of these are in wide use. Each method will be described briefly and its action discussed.

Fig. 1.15(a) Scoop sampling.

Fig. 1.15(b) Cone and quartering.

Fig. 1.15(c) Table sampler.
1.6.1 Scoop sampling (figure 1.15(a))

The method consists of plunging a scoop into the batch and withdrawing a sample. This is particularly prone to error since the whole of the sample does not pass through the sampling device, and since the sample is taken from the surface, where it may not be typical of the mass. In an attempt to eliminate segregation produced in previous handling, it is usual to shake the sample vigorously in a container before sampling.

Figure 1.16 shows five modes of shaking which were investigated by Kaye [8, 4], using six operators in order that both operator and technique bias could be evaluated. The samples consisted of coarse and fine particles mixed in known proportions.

Fig. 1.16 Five modes of shaking a container prior to removing a sample for analysis.

The various models were as follows:

1) In the vertical plane the bottle is moved rapidly around a circle. Superimposed on this motion is a brisk horizontal shaking in the direction of the arrows.
2) Brisk shaking in the vertical and horizontal planes; no swirling action.
3) The bottle is placed in the palm of the hand and then shaken in the manner shown with a brisk jerky action.
4) Starting in a vertical position the bottle is rotated to the right until it is inverted and then the rotation is reversed to the left until the bottle is again inverted. The cycle was carried out in two seconds.
5) The bottle is gripped firmly and then shaken in the direction of the arrows.
The first and third methods introduced no bias and the others a definite bias thus demonstrating that the efficiency is dependent on the previous history of the powder. Kaye also tested a standard laboratory shaker ‘the microid pipette flask shaker’ (Griffin and George Ltd), and found that its efficiency depended on its speed but even at peak efficiency it was no better than manual shaking.

1.6.2 Coning and quartering (figure 1.15(b))

This method of sample dividing consists of pouring the material into a conical heap and relying on its radial symmetry to give four identical samples when the heap is flattened and divided by a cross-shaped metal cutter (figure 1.15(b)). This method would give reliable results if the heap were symmetrical about a vertical axis and if the line common to the two cutting planes coincided with this axis. In practice the heap is unlikely to be symmetrical, and the second condition, symmetry of cutting, would be very difficult to achieve without precision equipment. Since severe size segregation will certainly occur in forming the heap, departure from symmetry in the cutting will lead to differences in the size of the four portions into which the heap is cut. The method is very dependent on the skill of the operator, and should not be used. If coning and quartering is possible, this implies that the quantity of material to be divided is such that it can easily be moved by hand; it is just as easy to feed it into the hopper of a device such as a rotary sample divider in which increments are collected from a stream in an acceptable manner.

1.6.3 Table sampling (figure 1.15(c))

In a sampling table the material is fed to the top of an inclined plane in which there are series of holes. Prisms placed in the path of the stream break it into fractions. Some powder falls through the holes and is discarded, while the powder remaining on the plane passes on to the next row of prisms and holes, and more is removed, and so on. The powder reaching the bottom of the plane is the sample. The objection to this device is that it depends on the initial feed being uniformly distributed, and complete mixing after each separation, a condition not in general achieved. As it relies on the removal of part of the stream sequentially, errors are compounded at each separation, hence its accuracy is low [4].

1.6.4 Chute splitting (figure 1.15(d))

The chute splitter consists of a V-shaped trough along the bottom of which is a series of chutes alternately feeding two trays placed on either side of the trough. The laboratory sample is poured into the chute and repeatedly halved until a sample of the desired size is obtained.

When carried out with great care on a laboratory scale this method can give satisfactory sample division, but if the trough is filled in such a way that segregation occurs the results are liable to be misleading. The method is particularly prone to operator bias, which is frequently detectable by unequal splitting of the sample.

In a review of factors affecting the efficiency of chute riffles Batel [6] stated that there is little to be gained by increasing the number of chutes. Kaye [4] points out that this is only true if the chute widths are kept constant; increasing the number of chutes by reducing the chute widths will increase the efficiency. He tested this hypothesis, using four riffles of the same overall width having 4, 8, 16 and 32 chutes, and found that the efficiency increased as the number of chutes increased.

It follows that two narrow, oppositely directed chutes intercepting the flow of powder would provide a very efficient sampling device. An equivalent selection procedure is to have one narrow powder stream oscillating between two reception bins. This in fact forms the basis of the British Standards oscillating hopper sample divider [7] (figure 1.17) and the ICI oscillating paddle divider (figure 1.18).

In the oscillating hopper sample divider, the feed hopper is pivoted about a horizontal axis so that it can oscillate during emptying.

![Oscillating hopper](image)

**Fig. 1.17 Oscillating hopper sample divider.**

Two collectors are placed under the hopper outlet so that the powder falls into them alternately. The contents of one box are retained; at each step the weight of sample is thus halved.

Kaye [4] suggests that samples of a given size may be obtained directly by controlling the amount of time, during each oscillation, that the feed is directed to each hopper or container. For efficient sampling the number of increments should be high.
1.6.5 The spinning riffler (figure 1.15(e))

The rotary sample divider or spinning riffler was first described in 1934 [9] and conforms to the golden rules of sample dividing. The preferred method of using this device is to fill a mass flow hopper, in such a way that little segregation occurs, by avoiding the formation of a heap (figure 1.19). The table is then set in motion and the hopper outlet opened so that powder falls into the collecting boxes. The use of a vibratory feeder is recommended to provide a constant flow rate. In 1959 Pownall [10] described the construction and testing of a large laboratory spinning riffler and a year later Hawes and Muller described the construction of a small instrument [11]. In 1964 Allen [12] described how six operators carried out identical experiments using three techniques. Little variation between operators is found with the spinning riffler but two-fold differences occur with chute splitting and cone and quartering. Several other investigations have also been carried out (see section 1.10).

The turntable type of sampler recommended in BS 3406 is very similar to the spinning riffler and a commercial version is available from Pascall (figure 1.20). In this instrument the powder falls through a hopper outlet on to a cone whose position may be varied to vary the outlet aperture. The powder slides down the cone into containers on a revolving table.
1.7 Miscellaneous devices

The moving-flap sample divider (figure 1.21) consists essentially of a flap which is pivoted about a horizontal axis so that it can rest in either of two positions. The stream of powder to be divided either falls down the process line to store or is diverted to the sampling section. The time during which the powder is sampled can be varied by varying the time during which the flap is in each of the positions and this may be done automatically. Since the diverted stream is still too large it is further divided on a table sampler and most of it is returned to store. Although the moving flap forms an efficient sampling device the table sampler does not, hence the combination will give a biased sample.

A splitter described by Fooks [13] (figure 1.22), consists of a feeder funnel through which the sample is fed. It passes on to the apex of two resting cones, the lower fixed, the upper adjustable by means of a spindle. Segments are cut from both cones and by rotation of the upper cone the effective area of these slits may be varied to vary the sampling proportion. Material which falls through the segmental slots is passed to a sample pot. The residue passes over the cone and out of the base of the unit.

A similar unit may be installed in a feedpipe down which particulate material is flowing. In this unit the splitter cones are mounted within the feedpipe and the sample falling through the segmental slots passes out of a side pipe, while the remainder flows over the cone and continues down the feedpipe.

Fig. 1.20 Rotary sample divider.

Fig. 1.21 Moving-flap sample divider.
Since the whole of the powder stream is not taken for many short increments of time this particular design is not recommended.

1.8 Reduction from laboratory sample to analysis sample

The methods described so far are suitable for the reduction of a gross sample collected from a process to a weight that can conveniently be sent to the size analysis laboratory, normally of the order of one kg. To obtain the amount required for the actual measurement a further reduction is necessary, and again careful consideration must be given to the problem of obtaining an analysis sample with the same size grading as the laboratory sample. The amount of material required as an analysis sample depends on the size measurement technique being used.

For sieving it would normally be 25 to 50 g; for sedimentation it would depend on whether the technique was cumulative (0.5 g) or incremental (8 – 12 g); for elutriation 0.5 g.

It is sometimes suggested that a satisfactory sample division can be made by shaking the bottle containing the laboratory sample in order to mix it, then extracting the analytical sample with a scoop [4, 8]. For accurate work this method should not be used for the following reasons:

1. Shaking a bottle containing a powder is not an effective method of mixing particles of different sizes. Anyone who doubts this should try the experiment of putting into a glass bottle equal volumes of two powders of different sizes and colours and examining the mixture after shaking. Pockets of segregated material form and cannot be broken up by further shaking. In particular the surface will be made up predominantly of the larger particles. A sample removed by a scoop must include part of the surface, where the composition is very likely to be different from that of the whole of the sample.

2. A further objection to the use of a scoop is that it is liable to be size-selective, favouring the collection of fine particles. The reason for this is that, when the scoop is removed from the material, some particles will flow down the sloping surface of the powder retained in the scoop; the finer particles tend to be captured in the surface craters and retained, whereas coarse particles are more likely to travel to the bottom of the slope and be lost. This effect is particularly important if a flat blade (such as a spatula) is used for the removal of the sample. Such methods should never be used.

The spinning riffler is the equipment recommended for subdivision down to a gram, but the powder needs to be free-flowing. Coning and quartering a paste [14] has been used for preparing samples down to about 20 mg in weight but the efficiency of the method is operator-dependent. Sampling a stirred suspension with a syringe is quite a good method for fine powder suspensions, but with suspensions of coarser powders, concentration gradients and segregation of particle sizes due to gravitational settling and the centrifugal motion of the liquid caused by the stirring action are more likely to arise. For these reasons Burt et al. [15] have developed a suspension sampler which is shown in figure 1.23. The sampler consists of a glass cylinder closed at either end by stainless-steel plates. Around the periphery of the base plate are ten equidistant holes leading to ten centrifuge tubes via stainless-steel capillary tubes. The cover plate has a central hole through which passes a stirrer, a sealable inlet for introduction of the powder suspension, and a gas orifice which enables the gas pressure to be increased or decreased. In operation 100 cm³ of suspension are introduced, stirred, then blown into the test tubes under a small applied pressure. Burt analysed a mixture of barium sulphate (0.25 g) and calcium carbonate (0.25 g) in 100 ml of water. The percentage of barium sulphate in each of the ten tubes was then determined and found to give significantly better results than syringe withdrawal, i.e. approximately 1.0% standard deviation as opposed to 3.0%.
Lines [31] compared three methods of sampling prior to Coulter analysis and found a spread of results of 6% with cone and quartering dry powder, a standard deviation of ± 3.25% with random selection and ± 1.25% with coring and quartering a paste.

Fig. 1.23 Burt's suspension sampler. (a) Suspension sampler, 10 ml to 2 ml

1.9 Reduction from analysis sample to measurement sample

In microscopy the required sample consists of a few milligrams of material. This may be extracted from the analysis sample by incorporating it in a viscous liquid in which it is known to disperse completely. The measurement sample may then be extracted with a measuring rod [7]. An alternative technique is to disperse the analysis sample in a liquid of low viscosity with the addition of a dispersing agent if necessary. The measurement sample may be withdrawn using a pipette or, preferably, a sample divider as illustrated in [16].

1.10 Experimental tests of sample-splitting techniques

In 1960 Hawes and Muller critically [11] examined the spinning riffler to find how various factors influence its efficiency. They used quartz and copper sulphate crystals of the same size and designed experiments to investigate the relative effect of the different factors. Their conclusions were that the efficiency:

(a) Is dependent on the relative proportions of the mixture (it increased when the proportion of copper sulphate was raised from 1% to 5%).
(b) Increases with increasing particle size.
(c) Is reproducible under similar experimental conditions.
(d) Is not affected by combination of variables.
(e) Is not affected by the number of volume units (a volume unit consists of the single presentation of a sample container to the feed. Since the minimum number of such units was 100, this statement applies only to larger values than this).

The powders mixed in these experiments contained particles all of the same size, hence the main cause of segregation was not present, and the conclusions may not apply in the more usual case where size segregation occurs.
In 1968 Khan [17] investigated the relative efficiencies of five common sampling procedures: scoop sampling, cone and quartering, table sampling, chute riffling and spinning riffling. Binary mixtures of different size ranges and different densities were sampled and the results examined statistically.

The results for the binary sand mixture are presented in Table 1.1 [18].

<table>
<thead>
<tr>
<th>Method</th>
<th>Standard deviation of samples (%)</th>
<th>$\text{Var}(P_n)$ (%)</th>
<th>Estimated maximum sample error (%)</th>
<th>Efficiency (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cone and quartering</td>
<td>6.81</td>
<td>46.4</td>
<td>22.7</td>
<td>0.013</td>
</tr>
<tr>
<td>Scoop sampling</td>
<td>5.14</td>
<td>26.4</td>
<td>17.1</td>
<td>0.022</td>
</tr>
<tr>
<td>Table sampling</td>
<td>2.09</td>
<td>4.37</td>
<td>7.0</td>
<td>0.13</td>
</tr>
<tr>
<td>Chute riffling</td>
<td>1.01</td>
<td>1.02</td>
<td>3.4</td>
<td>0.56</td>
</tr>
<tr>
<td>Spinning riffling</td>
<td>0.125</td>
<td>0.016</td>
<td>0.42</td>
<td>36.3</td>
</tr>
<tr>
<td>Random variation</td>
<td>0.076</td>
<td>0.0058</td>
<td>0.25</td>
<td></td>
</tr>
</tbody>
</table>

It was deduced that very little confidence can be placed in the first three techniques and the spinning riffler was so superior to all other methods that it should be used whenever possible.

Since the spinning riffler was far superior to the other techniques this was examined further using factorial design experiments in order to determine its optimum operating conditions. It was found that a minimum of 35 presentations is required to give optimum results. If the speed of rotation is made too great the efficiency will fall again since powder will be lost due to the air currents set up by the spinning disc.

In 1968 a comparison between the spinning riffler and the Jones chute splitter indicated that the latter introduced bias into the samples [19]. This was later confirmed by Kaye [4].

Several other articles [19 - 24] conclusively prove the superiority of the spinning riffler over all other methods investigated, and this method is recommended in ASTM Standard F577B - 78.

In 1978 Hatton [23] deduced theoretically that $\text{Var}(P_n) \propto \left(1 + \frac{L}{V}ight)^{1/2}$ where L is the linear flow rate of the feed and $V$ the peripheral velocity of the spinning disc. He found that the equation fitted Allen's data, which suggests that in order to reduce variations it is necessary to reduce $L/V$ which may be effected by reduction of $L$. 

References
8. SBS 3406 (1961): *Methods for the Determination of Particle Size of Powders, Part 1, Sub-Division of Gross Sample Down to 0.2 ml*.
2 Sampling of dusty gases in gas streams

2.1 Introduction

Legislation requires that manufacturers carefully monitor and control their particulate gas discharge effluent. Pollution today is a very 'dirty' word and visible discharge from chimneys with deposition of soot and sulphurous compounds creates a barrier between the industrialist and an environment-conscious public. The industrialist who accepts his duty to society and the environment equips his factories with dust-arresting plant such as filters, cyclones, electrostatic precipitators and scrubbers, balancing the cost against his profit margins. His less public-spirited counterpart keeps this non-productive plant to the minimum required by law. In general, financial and technical resources and an incentive to act are necessary for results.

The dust-generating industries have shown little incentive to devote resources, or spontaneously show any resolve to control, except where such control could be directly reflected for economic reasons [1].

The main economic incentives to control and measure are to reduce production losses and production costs. Government health inspectors must also be satisfied and the risk of successful legal actions minimized.

In the UK, the Public Health Act (1936) can be invoked. Health is defined as a state of well-being, both physiological and psychological, and the concept of a healthy environment includes the protection of inanimate objects from damage.

It is therefore necessary that dusty gas streams be regularly sampled to ensure both that the plant is working efficiently and that legal requirements are being met. Procedures for sampling particulate matter suspended in a gas stream have been described [2-8, 75]. These all emphasize the need to obtain representative samples. The samples must be taken across the cross-section of the duct to represent the total dust burden. The size distribution is usually required so that the deposition pattern of the discharge may be estimated, the larger particles falling more rapidly than the smaller ones. Since the particles are very fragile, the sampling device often incorporates size-grading sections. Representative samples are extremely difficult to obtain, a single series of tests may well take a week or more and a complete survey on a large plant may take several months.

It is generally accepted that any obstruction to flow will create turbulence and an asymmetrical dust burden (figure 2.1), but even in the absence of obstacles the dust concentration is non-uniform [9]. It is therefore imperative to sample at many points over the cross-section.

In vertical pipes the concentration can be assumed axisymmetrical and it is permissible to take samples along one radius of cross-section only; in other cases it is necessary to cover the whole cross-section [10]. Problems are also encountered in gaining access to the gas stream [11, 12].

There are then difficulties in removing a sample which is representative of the gas stream at this point, since a sample will be representative only if the velocity of aspiration into the nozzle is equal to the gas velocity at that point. This is known as isokinetic
sampling. Under isokinetic conditions there is no disturbance of the gas streamlines and all the particles approaching the nozzle, and only those, will enter it [13 - 16].

In order to obtain representative samples it is also necessary that the sampling probe should be parallel to the flow lines (isoxial), otherwise a loss of concentration occurs.

Walter [17] asserted that, for representative sampling, the sampling velocity should be greater than the gas velocity in the duct. This was later disproved and Walter's results were shown to be due to the use of sampling probes with thick walls and obtuse angles [10].

Rouillard and Hicks [70], on the basis of experimental work, suggest that for a sampling system to cause negligible disturbance of the velocity profile, sampling should be isokinetic using a knife-edge nozzle with little external bevel and the stem of the probe should be at least eleven diameters downstream of the nozzle.

Dust that settles in the probe should be collected and a gravity correction should be applied if the duct is vertical, but this is usually not done due to the complexity of the correction.

As it is not possible to collect increments of the whole emission it is necessary to select suitable sampling positions across the cross-sectional area perpendicular to the direction of gas flow. Since the quantity of emission varies with time, results can only give an average for the time of tests. Tests should be dispersed and made averages taken. If sampling is carried out in accordance with BS 3405 a minimum of four points are sampled, each for a minimum duration of two minutes. It is necessary to ensure that the actual sampling time is adequate to give a quantity of dust which can be accurately weighed. Agreement should be better than 50% and the average taken [2]. Increasing the number of points to eight should improve the likely accuracy to 15% and an increase to 24 with a sampling time of ten minutes should give an accuracy of 5% [3].

Greater discrepancies may indicate a faulty test or that plant conditions have varied, hence further tests are required. The plant cycle should also be observed to establish loading conditions. Averages do not normally include short periods of high dust emissions during soot blowing. Attempts to include these could well render it impossible to get any measure of agreement.

When sampling hot aerosols containing condensible vapours it is necessary to have a heated box for an external filter otherwise the vapour condenses and clogs the filter. Removal of the vapour can lead to dust losses.

When the gas to be sampled is at a temperature in excess of 400°C problems arise in the selection of sampling equipment which is often not suited for such extreme conditions. For example, pitot-static tubes, though manufactured from stainless steel, have silver-soldered joints [71]. One solution is to use water-cooled tubes and probes.

These problems are circumvented with the use of internal sampling and replaced by the problems of corrosion and the difficulty of installing bulky equipment in the duct without interfering with flow. On the whole, external sampling is preferable [18, 19].

A wide range of duct velocities can be encountered; hence, in order to maintain isokinetic conditions, a series of nozzles of different diameters is necessary. These are particularly useful when the sampling volume flow rate can only be adjusted over a limited range or when the collecting device exhibits a constant pressure drop.

### Table 2.1: Mean values of relative particle concentrations (radius of bend, 2D; gas velocity, 40 ft s⁻¹)

<table>
<thead>
<tr>
<th>Number of diameters from bend</th>
<th>A1</th>
<th>A2</th>
<th>A3</th>
<th>A4</th>
<th>B1</th>
<th>B2</th>
<th>B3</th>
<th>B4</th>
<th>C1</th>
<th>C2</th>
<th>C3</th>
<th>C4</th>
</tr>
</thead>
<tbody>
<tr>
<td>H</td>
<td>0.66</td>
<td>0.72</td>
<td>0.51</td>
<td>0.84</td>
<td>0.64</td>
<td>0.56</td>
<td>0.53</td>
<td>0.90</td>
<td>0.65</td>
<td>0.83</td>
<td>1.19</td>
<td>1.12</td>
</tr>
<tr>
<td>1</td>
<td>0.33</td>
<td>0.24</td>
<td>0.20</td>
<td>0.78</td>
<td>0.35</td>
<td>0.15</td>
<td>1.35</td>
<td>2.13</td>
<td>0.27</td>
<td>0.22</td>
<td>0.13</td>
<td>0.68</td>
</tr>
<tr>
<td>2</td>
<td>0.49</td>
<td>0.43</td>
<td>1.01</td>
<td>0.34</td>
<td>0.15</td>
<td>0.98</td>
<td>1.42</td>
<td>0.25</td>
<td>0.37</td>
<td>0.76</td>
<td>1.46</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>0.67</td>
<td>0.47</td>
<td>1.13</td>
<td>0.28</td>
<td>0.26</td>
<td>0.57</td>
<td>1.15</td>
<td>0.33</td>
<td>0.38</td>
<td>0.76</td>
<td>1.28</td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>0.78</td>
<td>0.59</td>
<td>0.73</td>
<td>0.73</td>
<td>0.73</td>
<td>0.56</td>
<td>0.70</td>
<td>0.62</td>
<td>0.57</td>
<td>0.71</td>
<td>0.79</td>
<td></td>
</tr>
<tr>
<td>16</td>
<td>0.78</td>
<td>0.59</td>
<td>0.73</td>
<td>0.73</td>
<td>0.73</td>
<td>0.56</td>
<td>0.70</td>
<td>0.62</td>
<td>0.57</td>
<td>0.71</td>
<td>0.79</td>
<td></td>
</tr>
</tbody>
</table>

H indicates the horizontal cross-section; A is a diameter through the elbow of the bend; B and C are at 30° to diameter A.
Sansone [5] investigated the patterns of flow of solids downstream from a 90° bend. Particles were fed into an airstream in a horizontal duct and concentrations were determined in a vertical section using a twelve-point sampling pattern at the centre of annuli of equal areas. Some of his results are given in Table 2.1. The data in the above table reveal that concentrations are generally higher immediately downstream of a bend along the duct wall farthest from the centre of curvature of the elbow. Similarly the concentrations are low along the duct wall nearest the centre of turning. In negotiating the bend, particles resist change of direction and are thrown to the outer wall. There is also a loss in concentration due to assuming that particle concentrations at centres of equal area are representative of these areas. These effects are enhanced with a gas velocity of 80 ft s⁻¹.

2.2.2 Temperature and velocity surveys

These should be carried out at ten equally spaced points along a sampling line, excluding the 50 mm nearest the flue walls. If temperature variations exceed 10%, a leak

Fig. 2.2 Dust arrester on test; laboratory rig.

Sampling of dusty gases in gas streams

should be suspected and this should be plugged otherwise the position is unsuitable.

If the ratio of maximum to minimum velocity exceeds 2, the four-point method of sampling is unsuitable. Gas flow is rarely uniform across the cross-section of a duct. For streamline flow (Re less than 2000), the velocity on the axis is twice the mean velocity, and for fully turbulent flow (Re greater than 2500), the axial velocity equals the mean velocity at D/6 from the walls, and it is claimed that a sample extracted at this point will be representative, but this is discounted by Stairmand [4], who states that the dust distribution rarely follows the gas distribution. Hawksley [20] also discusses variation of dust and gas flow across a duct.

2.2.3 Sampling points

In BS 893 the use of 24 sampling points, with at least three access holes, is recommended. The sampling lines should form the diameters of a hexagon with sampling access at alternate ends and sampling points at 0.07r, 0.21r, 0.39r, 0.65r, 1.35r, 1.61r, 1.79r, 1.94r from the access point (r being the radius of the flue). These points are at the centres of annuli of equal area. Rectangular ducts should be divided up into 24 rectangles of equal area with sampling points at the centres of each rectangular element.

In BS 3405 four or eight points are recommended, again at the centres of annuli of equal area. The four points lie on mutually perpendicular diameters at a distance 0.7r from the centre of the duct. With eight sampling points, four lie on mutually perpendicular diameters a distance 0.5r from the centre of the duct and four on the same diameters 0.87r from the centre of the duct. A reduction to a single sampling point has been made by Stairmand (figure 2.2) [4, 34, 38, p. 583] who uses a half-area centrally located obstruction (a mixing baffle), three duct diameters upstream of the sampling probe, leaving at least two duct diameters of straight pipe downstream of the sampling probe. He claims that the sampling baffle makes the concentration of dust at the sampling position fairly uniform.

A similar device, the ring and doughnut flow straightener, has also been described [11]. Fuchs [10] says of Stairmand's half-area baffle: 'a considerable part of the dispersed phase will be deposited on the screen and afterwards will pour down or be blown off the screen. The method is useless for particle size determination but may, under favourable conditions, measure concentration.'

A device for routine sampling, for comparative purposes only, has also been developed [22] (figure 2.3). This consists of a suitable probe inserted into the flue duct. The open end faces the gas stream where it flows either horizontally or downwards so that the momentum of the grit carries it into the sampler. The device was used to detect failure of electrostatic precipitators and multicyclones installed on boilers earlier than would otherwise have been possible without expensive testing.

2.3 Sampling equipment

The accepted method of sampling flue gases in the UK is given in BS 893 [3]. The method is lengthy and complicated, but in spite of development and experience, more recently issued standards of other countries [23] differ only in detail. A more recently
Sampling of dusty gases in gas streams

issued British Standard (BS 3405) [2] gives simplified procedures for quick investigations.

The basic equipment consists of a set of nozzles attached to a sampling tube; a miniature collector to retain the particles; means for determining the amount of gas sampled and the gas velocities in the ducts; means for withdrawing samples of gas through the system; thermometers, manometers, cooling coils, catchpots, connecting tubing and stopwatches.

(a) Section showing requirements at open end of a sampling nozzle

(b) Diagram showing clearances necessary between open end of sampling nozzle and other parts of the apparatus

Fig. 2.4 Requirements for sampling nozzles (BS 3405).
2.3.1 Nozzles [3, 4, 12]

The nozzles may vary in shape and construction but they should be of circular cross-section, the open end having a thickness less than 1.3 mm and the internal and external surfaces having an inclination not greater than 45° to the axis of the nozzle (figures 2.4 and 2.5). With blunt-edged probes, a damming effect occurs upstream from the probe which deflects particles away from it [24 – 28]. The nozzle size must be chosen so that, with isokinetic sampling, the maximum flow rate will be within the capacity of the sampling equipment. Tables showing the correct nozzle size for the maximum pitot differential are usually supplied with the dust-sampling apparatus.

When using these simple sampling nozzles the flow field must be previously measured with a pitot tube.

An alternative solution is to use a pump-free system. The Cegrill (figure 2.6) is an example of this type of device [19]. The device consists of a heated cyclone through which a sample is continuously drawn by an ejector device operated by the suction of the flue itself, the dust collecting in a detachable glass container. The great ingenuity of the device lies in its continuous isokinetic operation irrespective of variations in gas flow.

4 fixing positions to suit direction of flow

Fig. 2.5 Alternative designs for sampling nozzles.

Null-type nozzles have double walls in which there are two ring-like channels connected by holes with the inside and outside of the probe. For isokinetic sampling the pressure difference in these channels should be zero (see figure 2.7) [29 – 32].

Velocity-sampling nozzles are a combination of sampling probe and pitot tube. The flow velocity is determined by the difference between the dynamic and static pressure. A drawback is the clogging of holes at high dust concentration. These probes were extensively studied by Noss [33].

Rammler and Breitling [18] examined the various available systems and preferred the velocity-sampling probes.
2.3.2 Dust-sampling collector

The purpose of the sampling collector is to remove the particulate matter from the gas sample extracted from the main duct. Where it is only desired to determine concentration, the choice is fairly easy; where composition or size distribution has to be determined, more precautions have to be taken. Stairmand describes a range of collectors in [4] and discusses their performance; other descriptions are to be found in [2].

The perfect collector would offer little resistance to gas flow even when it had collected a considerable amount of dust. Further, the dust should be easily separable from the collector without residual contamination. The only collector without these requirements is the electrostatic precipitator, but difficulties arise in the use of this equipment [34].

The range of collectors described by Stairmand consists of:

(a) Cyclone separators.
(b) Glass-wool filters.
(c) Ferrule glass-wool filters.
(d) Slag-wool filters.
(e) Composite filters.
(f) Terylene and superfine glass-wool filters.
(g) Soluble filters.
(h) Volatile filters.
(i) Soxhlet filters.
(j) Ceramic thimble filters.
(k) Electrostatic filters.
(l) Impingement filters.

A high-efficiency cyclone (figures 2.8 and 2.9) forms a useful sample collector, since it can collect large quantities of dust without increase of resistance. In many cases it will trap more than 95% of the suspended matter and may be used without a backing filter. When the dust is fine or complete removal is required, the cyclone may be backed by any of the range of filters annotated above.

A typical 'absolute' filter is shown in figure 2.10, an electrostatic filter in figure 2.11 and an impingement filter in figure 2.12.
Pitbl et al. [35] described a cascade impactor which is inserted into the stack in series with an inlet nozzle and filter assembly. Bridger [71] describes the instrument in use and prefers it to the Andersen sampler (see figure 2.15).

The simplest equipment consists of a small cyclone without a series filter. Locating the cyclone near the nozzle end of the probe so that it is within the flue, is a further simplification, since the need to heat the cyclone and probe to prevent condensation is eliminated. The sampling rate and gas volume may be determined by measuring the pressure drop across the cyclone or by means of an orifice plate.

A refinement of the above apparatus is to pass the gas from the cyclone through a filter, usually of packed glass fibres, thus permitting the collection of particles that have escaped through the cyclone [36, 37]. Proprietary equipment based on the above design are used by BCURA [42], CEGB, NCB and others [38, p. 604].

Other equipment has heated filters on the outward end of the probe or unheated filters on the inward end of the probe [25]. Equipment developed by BISRA for rapid sampling using filters is described by Granville and Jaffrey [36].

The IC sampler forms one of the most useful of the composite equipment [4] (figure 2.13). It consists of a nozzle attached to a heated probe, glass-wool filter, cooling coil, catchpot, orifice plate flowmeter and an ejector. Appropriate manometers and thermometers are provided so that the necessary calculations for correct isokinetic sampling can be made.

![Diagram of equipment for sampling dust from flowing gases.](image)

With the CEGB Mark II dust sampler the dust is collected in a glass fabric or paper thimble. Details of design and operation are given in [38, p. 605].

With the impingement-type filters, the dust is classified according to its aerodynamic properties. This is preferable to microscopic methods or sieving since these properties may more easily be related to particle behaviour on leaving the stack, the approximate area of deposition and the probable point of respiratory deposition. Further, the classification is carried out in situ which reduces the probable breakdown of floculates.

The Day isokinetic dust sampler is used to measure dust particles discharged for dust control equipment as well as dust in dust control ducts. It is basically a thimble and a nozzle with a rotameter and pump.

With the Staksampler, sampling is isokinetic with the sample passing through a glass sampling train where the particulates and gases are collected.

The sample from the Dust Difficulty Determinator is drawn, by means of an exhaust fan, through two wet scrubbers. The first scrubber has a pressure drop of 5 in water and the pressure drop in the second can be varied over the range 5 to 100 in water.

Farthing [21] recently reported on a conference at which seventeen papers were presented on improved instruments and techniques for measuring the size, number and composition of particles in process streams.

Recent studies were presented on cascade impactors, cyclones and diffusion batteries and several methods of making in situ size or mass determination by light scattering were reported.

### 2.3.3 Ancillary apparatus

(a) Pitot tube (BS 1042). This should face directly into the gas stream with a maximum deviation (angle of yaw) of 10°.

(b) Pressure-measuring instrument(s). An inclined gauge reading 0.05 to 0.30 mbar within ± 0.01 mbar.

(c) A probe tube to which nozzle is attached maintained at a temperature sufficient to prevent condensation. If the dust collector is fitted immediately after the nozzle and before the probe, this requirement does not hold. In the former case the probe should be constructed of stainless steel.

(d) Flow and temperature-measuring device to measure ν to 5% and T to 5 K.

(e) A control valve to regulate flow through the equipment.

(f) A method for withdrawing sample at required rate for isokinetic sampling.

### 2.3.4 On-line dust extraction

An on-line isokinetic particle sampler for continuously sampling finely divided materials being transported in a flow system is the subject of a patent [39] (figure 2.14). The particulate material flows through conduit (1) and is sampled at nozzle (2), which is positioned high enough into the flow stream so that it is not affected by any disturbances in the flow due to housing (3). An artificial atmosphere of dry nitrogen gas is maintained within sampling tube (4) to control the pressure within the tube as well as to transport particles out of it. The nitrogen exits at (5) creating a partial vacuum at the intersection with (2), thus drawing the particulates into the system isokinetically. The pressure is maintained at the pressure in conduit (1) by outlet (6), which is con-
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connected to the automatic differential pressure control (4). When a difference in pressure is noted, a signal is fed to the automatic valve which varies the valve position to maintain equal pressure.

![Diagram of particle size measurement system](image)

**Fig. 2.14** Isokinetic particle sampler (Lynn [39]).

2.3.5 *The Andersen stack sampler*

The Andersen stack sampler [40] has a cascade impactor-type sampling head which, in operation, is mounted inside the flue (figure 2.15).

The sampler contains nine jet plates, each having a pattern of precision-drilled orifices. The nine plates, separated by 2.5 mm stainless-steel spacers, divide the sample into eight fractions of particle size ranges. The jets on each plate are arranged in concentric circles which are offset on each succeeding plate. The size of the orifices is the same on the given plate, but is smaller for each succeeding downstream plate. Therefore, as the sample is drawn through the sampler at a constant flow rate, the jets of air flowing through any particular plate direct the particulates toward the collection area on the downstream plate directly below the circles of jets on the plate above. Since the jet diameters decrease from plate to plate, the velocities increase such that whenever the velocity imparted to a particle is sufficiently great, its inertia will overcome the aerodynamic drag of the turning airstream and the particle will be impacted.

![Diagram of Andersen stack sampler](image)

**Fig. 2.15** The Andersen stack sampler.
on the collection surface. Otherwise, the particle remains in the airstream and proceeds to the next plate. Since the particle deposit areas are directly below the jets, seven of the plates act as both a jet stage and a collection plate. Thus, No. 0 plate is only a jet stage and No. 8 plate is only a collection plate.

The Andersen stack sampler has been calibrated by several independent laboratories in order to arrive at the correct respective size cuts for each stage. The calibrators are referenced to unit density (1 g cm\(^{-3}\)), spherical particles, so that the aerodynamically equivalent-sized particles collected on each stage are always identical for any given flow rate. For this reason, a stack sample containing a mixture of shapes and densities is fractionated and collected according to its aerodynamic characteristics and is aerodynamically equivalent in size to the unit density spheres collected on each specific stage during calibration. A seven-stage Andersen impactor has been used for soot blowing and for non-isokinetic as well as isokinetic sampling [72].

2.4 Corrections for anisokinetic sampling

A measure of dust content can be expressed as:
(a) The mass flow rate of dust. This is the mass of dust passing per unit time across an element of area of the gas stream. In isokinetic sampling, the quantity directly determined is the amount of dust passing, during the time of sampling, across an area in the gas stream equal to the area of the nozzle opening.
(b) The concentration of dust. This is the mass of dust per unit volume of gas, and is usually calculated from the ratio of the mass of dust collected to the volume of gas sampled. When sampling is isokinetic, the concentration of dust in the sample is equal to the concentration in the gas stream at the point of sampling.

When the sampling velocity differs from that of the gas stream (anisokinetic sampling), the gas streamlines are disturbed, causing some particles to be deflected from their original direction of motion, so that the quantity of particles entering the probe per unit time will differ from that entering under isokinetic conditions (see figure 2.16).

It will be less when the sampling velocity is less than that of the gas stream, because some of the gas that should enter the probe flows past it carrying some particles with it. Conversely, if the sampling velocity is higher than the amount of particles entering the probe will be too high. It is easy to envisage what happens in the case of very fine or very coarse particles. When the particles are very fine, they follow closely the deflected gas streams and the amount entering is proportional to the sampling velocity.

That is, the concentration of fine particles in the sample is equal to that of the gas stream, irrespective of the sampling velocity. When the particles are coarse, their inertia is so great that they persist in their original direction of motion; the amount entering the probe is independent of the sampling velocity. That is, the mass flow rate of coarse particles is measured correctly irrespective of the sampling velocity, a fact first reported by Hemeon and Haines [63].

If the particles cover a range of sizes, anisokinetic sampling affects not only the mass of particles in the sample, but also their size distribution. During prolonged sampling the flow rate of the gas stream may vary throughout the test, and it may not be possible to vary the sampling rate to compensate for this. A method for correcting this is described later.

![Fig. 2.16](image)

(a) Isookinetic sampling: representative concentration and grading.
(b) Sampling velocity too low; excess of coarse particles.
(c) Sampling velocity too high; deficiency of coarse particles.

Seldon [41] suggests that deviations from isokinetic sampling conditions give rise to errors in concentration which may be summarized in the general formula:

\[
A = 1 + \left( \frac{1}{w} - 1 \right) F
\]

where

\[
A = \frac{C_s}{C}
\]

\[
C_s \text{ is the solids concentration in the sampled gas}
\]

\[
C \text{ is the solids concentration in the gas stream.}
\]

\[
w = \frac{v_s}{v}
\]

\[
\nu_s \text{ is the sampling velocity}
\]

\[
v \text{ is the gas stream velocity}
\]

\[
F \text{ is a function of the dimensionless groups.}
\]

The Reynolds number \( Re = \frac{\rho_f v_d}{\eta} \)

where \( \rho_f \) is the fluid density,
\( \eta \) the fluid viscosity and
\( d \) the particle diameter.
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The Stokes number \( Stk = \frac{\nu_f D}{g D} \) (2.5)

where \( g \) is the acceleration due to gravity,
\( D \) is the nozzle diameter and
\( \nu_f \) is the free-falling velocity of the particle.
For \( Re < 0.5 \) the free-falling velocity may be replaced by the Stokes velocity.
Other terms in general use are:
\( \lambda \), the particle range, the distance the particle will travel in still air before coming to rest;
\( \tau \), the particle relaxation time in the gas.
\[ Stk = \frac{\lambda}{D} = \frac{\tau \nu}{D} \]  
(2.6)

From equations (2.5) and (2.6) For \( Re < 0.5 \)
\[ \tau = \frac{\rho_p d^2}{18 \eta} \]  
(2.7)
\( \rho_p \) is the particle density.

Badziach [26] stated that the mass of particles \( M_e \) entering the nozzle at a sampling velocity \( \nu_s \) is related to the mass \( M \) entering the nozzle when the sampling velocity \( \nu \) equals the gas velocity by the equation:
\[ \frac{M_e}{M} = (1 - \alpha)w + \alpha \]  
(2.8)
where:
\[ \alpha = 1 - \lambda \left( \frac{L}{\lambda} \right) \]  
(2.9)
\( L \) is a length representative of the distance upstream of the nozzle over which there is a disturbance in the gas stream [27].

---

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Badziach's work is questioned by Fuchs [10] who states that the scatter of \( A(\frac{C_2}{C}) \) values obtained by him is very large and differs considerably from later data.

A versus \( w \) values have been calculated by Vitols [42] at different Stokes numbers.
Isokinetic sampling is only critical in a narrow size range, the limits of which are conveniently expressed in terms of Parker's [43] inertia parameter \( \psi \)
\[ \psi = C Stk \]  
(2.12)
where \( C \) is the Cunningham correction factor.
Parker stated that isokinetic sampling was necessary for:
\[ 0.05 < \psi < 50 \]  
(2.13)

Svarovsky [44] shows the effect of sampling, nozzle diameter and flow velocity on the upper and lower limits of particle size, in graphical form, using Parker's inequality relationship.

Ruping [45] measured the flow field by means of a probe and calculated the resulting particle trajectories. His results deviate widely from the later experimental data.

Voloshchuk and Levin [46] based their work on earlier work by Vdovay [47] and, although they made some sweeping assumptions, they obtained reasonable agreement with experimental work.

The most relevant measurements were made by Zeuker [48] who found that:
\[ \frac{M_e}{M} = 1 + K(w - 1) \]  
(2.14)
for \( 0.4 < w < 2.5 \).
\( K \) is a coefficient depending only on the Stokes number and equal to 1 at \( Stk = 0 \) and to 0 at \( Stk = \infty \).
As \( M_e \) is proportional to \( C_2 \nu_s \) and \( M \) to \( C_2 \),
\[ A = K + (1 - K) / w \]  
(2.15)

Other experiments were performed by Belyasov and Levin [49, 50] and their results can be approximated to with the formula:
\[ A = 1 + \left( \frac{1}{w - 1} \right) \left( \frac{2 + 0.62w}{1 + (2 + 0.62w) Stk} \right) \]  
(2.16)
for \( 0.18 < w < 5; 0.18 < Stk < 2 \).
Their results are in fair agreement with Zeuker's [10].

Comparing Badziach's equation (2.8) with Seldon's general equation (2.1) gives \( F \) as a function only of \( Stk \). Equation (2.16) gives \( F \) as a function of \( w \) and \( Stk \). Seldon suggests that \( F \) is a function of three-dimensionless groups and proposes an equation similar to equation (2.16) which, rearranged, gives:
Particle Size Measurement

\[ N = \frac{Aw - 1}{w - Aw} \]  
(2.17)

\[ = f(S_t k, Re, w) \]  
(2.18)

For equation (2.16) \( a = 1, b = 2, c = 0.62, d = 0 \). Seldon proposes \( a = -0.918, b = 1.53, c = 0.617, d = 0.355 \).

The error due to anisokinetic sampling is determinable as follows: The true dust concentration in the gas stream is given by:

\[ C = \frac{M}{aw} \]  
(2.19)

where \( a \) is the area of the sampling nozzle. It follows that, when sampling fine dusts, the observed concentration will be nearly correct whatever the sampling velocity, provided it is calculated from the rate of dust collection \( M \) divided by the volume flow rate of sampling \( aw \):

\[ C_s = \frac{M}{aw} \]  
(2.20)

When sampling coarse dusts anisokinetically, a correct value is obtained if \( M \) is divided by \( aw \), i.e.:

\[ C_s = M_s / aw \]  
(2.21)

When the fine dust method is used, the percentage error \( E \) is given by:

\[ E = 100 \left( \frac{C - C_s}{C} \right) = 100 \left( A - 1 \right) \]

\[ = 100 \left( \frac{100}{N + 1} \right) \left( \frac{1 - w}{w} \right) \]  
(2.22)

Similarly when the coarse dust method is used, the apparent dust concentration is given by:

\[ E' = 100 \left( \frac{C - C_s}{C} \right) = 100 \left( \frac{w - 1}{N + 1} \right) \]  
(2.23)

The latter method will give more accurate results if \( E' \) is less than \( E \), if:

\[ N > \frac{\nu}{\nu_s} \]

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The magnitude of the errors involved in using these formulae can be obtained from Table 2.2. For example, with \( D = 1.25 \text{ cm}, \nu = 800 \text{ cm}^2 \text{s}^{-1}, \nu_s = 1600 \text{ cm}^2 \text{s}^{-1}, \lambda = 1.53 \text{ cm}^2 \text{s}^{-1} \) making \( \lambda = 1.25 \text{ cm} \) and \( S_t k = 1 \), the error is \( E = -38.2\% \) or \( E' = -76.4\% \).

### Table 2.2 Percentage errors in concentration (E), lower triangle and (E'), upper triangle, as a function of Stokcs number and relative velocity for anisokinetic sampling.

<table>
<thead>
<tr>
<th>w</th>
<th>0.2</th>
<th>0.3</th>
<th>0.5</th>
<th>1</th>
<th>2</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.2</td>
<td>4.8</td>
<td>119</td>
<td>2.1</td>
<td>206</td>
<td>242</td>
</tr>
<tr>
<td>0.5</td>
<td>3.2</td>
<td>31.6</td>
<td>13.4</td>
<td>53.6</td>
<td>34.9</td>
</tr>
<tr>
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<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
</tr>
<tr>
<td>2</td>
<td>-7.8</td>
<td>-19.6</td>
<td>-30.9</td>
<td>0.0</td>
<td>-76.4</td>
</tr>
<tr>
<td>5</td>
<td>-13.1</td>
<td>-144</td>
<td>-57.5</td>
<td>-338</td>
<td>-66.9</td>
</tr>
</tbody>
</table>

2.5 Probe orientation

An essential detail of isokinetic sampling is that the entry nozzle axis should be aligned with the wind direction (isoxial sampling). A sampling probe making an angle \( \theta \) with the wind direction (angle of yaw) suffers a diminution of effective area related to the cosine of the yaw angle.

May [51] considers that the efficiency of entry of very small particles, which follow the air flow, is unaffected by yaw whereas the efficiency of entry of large particles obeys the cosine relationship. He defines large as referring to particles that have sufficient inertia to ignore bends in the streamlines and considers that they would be about 100 \( \mu \text{m} \) in diameter at low airspeeds; he defines very small as less than 1 \( \mu \text{m} \) in diameter.

As regards entry efficiency, as distinct from collection efficiency, the effect of yaw is not very serious; even with large particles and an angle of yaw of 25° the loss is only about 10% and for 5 \( \mu \text{m} \) particles May showed that the error would be about 7% with a yaw angle of 45°.

In most instruments the particles must pass down a tube immediately after entry and an important effect of yaw is that they may then be impacted into the side of the duct facing the wind direction. Losses can be very serious; they are related to the yaw angle, the duct width and the stopping distance of the particle. If the product of the particle's stopping distance and the sine of the yaw angle is greater than the duct width then impact is likely to occur [52].

Watson's data [53] for non-isoxial sampling are considered by Fuchs [10] to be unreliable. According to Fuchs, the correction for small \( \theta \) is:

\[ A = 1 - \frac{4}{\pi} S_t k \sin \theta \]  
(2.24)
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2.6 Radiation methods [62]

For in situ measurements the absorption of visible light, gamma rays and acoustical energy has also been investigated. The most widely attempted techniques have used absorption of visible light [54 - 56]. The two problems associated with this type of device are that turbidity is a function of the projected surface area of particles within the light beam, whereas most requirements are for a mass-dependent technique, and that the light absorbed by a particle is not linearly related to size, i.e.:

\[
\text{turbidity} = k \alpha S_w L K
\]

where \(k\) is a shape factor,
\(c\) is the concentration of particles in the beam,
\(L\) is the length of the light path in the suspension,
\(S_w\) is the weight-specific surface of the particles in the beam,
\(K\) the extinction coefficient, is the ratio of the light cut off by the particles to the light which would be cut off if the laws of geometric optics held.

Despite these objections the technique is useful for continuous monitoring.

Several instruments for measuring particulate concentration and size in smoke stacks have been produced commercially but, due to the breakdown in the laws of geometric optics, they are only suitable for comparison purposes.

The Nebeto continuous smoke monitor is designed to measure the optical turbidity across a 24 in or 36 in circular stack.

In the Leeds and Northrop smoke sampler, the smoke is extracted from the stack and passed through a light beam.

The Bailey smoke/dust density transmitter measures the turbidity across a stack.

The Edison visibility monitor is a forward light-scattering photometer having a receptor core from 12° to 70° using an ultra-violet source.

Lucas et al. [65, 66] discuss some of the limitations of smoke recorders (opacimeters) and state that the inaccuracies could be reduced by the use of a steel tube to maintain alignment, by the use of Everclear windows [67] and the use of purging air to record the effective instrument zero. They also discuss an instrument for coarse dust measurement (impactor) so called because it measures the opacity of dust after it has settled by impaction. The opacimeter is referred to as the CERL Self-Checking Smoke Recorder (commercially available as the SEROP) and the impacimeter as the CERL Flue Dust Monitor Mark II.

The LTV monitor [69] combines laser illumination, TV imaging and signal-processing electronics to determine particle emissions in the range 0.2 to 10 \(\mu\)m. The machine is portable and shows good agreement with EPA filter trains for measuring mass loads.

Harris et al. [68] in a paper entitled ‘Turbulence Noise and Cross-Correlation Techniques Applied to Measurement in Stacks’ applied the expertise they had gained in developing an on-line sizing instrument to the problems of stack sampling. The instrument they describe monitors forward, 30° and 90° scattering and processes the signals representing random fluctuations and discards information in the form of mean values of source radiation variations, drift or optical surface degradation. The signals are stored on magnetic tape and analysed off-line using cross-correlation functions. The original instrument has a collimated white light source and an alternative using a laser source is also described.

The Gelman Stack Monitor is a system designed for industrial stack monitoring. It can be used to sample, measure and record automatically the mass of emitted particles from a stack or duct system. The gas from the sampling probe is drawn through a paper tape filter and the weight deposited determined using \(\beta\)-ray attenuation. The RAC Automatic Stack Monitor is similar in principle to the Gelman instrument.

Nuclear Measurements Corporation manufacture a series of filtration samplers which incorporate a variety of radiation detectors including a-scintillation, Geiger-Müller and a \(\alpha\) - \(\beta\)-proportional counters.

The MAP-1B is a moving filter monitor for the continuous monitoring of the radioactivity of airborne particulate matter using a filter paper and detecting system.

An alternative approach to light attenuation utilized gamma radiation from a nuclear source and a Geiger-tube detector [57]. Absorption of the energy is again a function of path length, concentration and absorption factor. In this case the absorption factor is proportional to the atomic number, hence the mass of the particles, provided they are reasonably uniform in chemical composition.

A further technique is the absorption of acoustical energy [58]. The response is influenced by individual particle characteristics and gas density and gives very poor sensitivity to particle concentration.

Continuous analyses of extracted particulates have also been investigated. Several of these are a direct measurement of the mass collected and the primary problem is related to representative sampling. A system based on an electrobalance has been used as a direct measure of mass. The extracted particulate is deposited on a collection medium, which is then automatically cut off, transferred to the balance and weighed. The approach is really semi-continuous and would have application primarily where high accuracy on variable particulate is required. A second technique for direct mass measurement is based on frequency of oscillation as a function of the mass of material deposited on the surface. The most widely investigated extraction approach is based on collection of the particulate on a filter medium and detection of the mass through absorption of low-energy \(\beta\)-particles [59]. Absorption of the energy is a function of the total mass collected and chemical composition of the sample. Indirect measurements based on electrical devices, light absorbance and light reflectance have been employed. The electrical devices measure current transferred by the particles from a charging zone to discharge collection plates [60, 61]. Light absorbance and reflectance have also been used.

The application of continuous monitoring instrumentation presents fundamental difficulties. In particular the techniques do not satisfy usual requirements. They may be used as process monitors after complete characterization of the system using manual methods. However, the engineer should be fully aware of the complexities introduced by the interaction between particulate properties and potential measurement approaches.
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3 Sampling and sizing from the atmosphere

3.1 Introduction

Sampling from the atmosphere is carried out mainly to monitor health and nuisance hazards. The air is full of particulates, even in a non-industrial environment, but these are not usually noxious. The unenviable task of the legislator is to balance the needs of industry to pollute the atmosphere against the public demand for clean air. In Britain, the Clean Air Act of 1956 requires the discharge of effluent into the atmosphere to be reduced to a minimum using 'the best practical means' and health inspectors have been appointed to enforce this nebulous legislation. In the United States the Environmental Protection Agency (EPA) standards are more precise which makes the inspectors' job easier but raises other problems. The EPA standards for ambient air are that the annual geometric mean for particulate matter should not exceed 75 μg m⁻³ and that the maximum concentration should not exceed 260 μg m⁻³ more than once a year. Now if one compares Mobile, Alabama (124 μg m⁻³, mean) with New York (105 μg m⁻³, mean) it would seem that New York should have less difficulty in meeting this standard than Mobile. However, the reverse is true, since Mobile has no particulate matter controls whereas all controls possible have been installed in New York [1].

The inapplicability of this type of blanket regulation is highlighted by the Los Angeles requirement that emission from any one source must not exceed 20 lb hr⁻¹. This cannot be met by States which have, for example, cement plants; indeed one would not expect the same standards to apply to heavy industrial areas as apply to areas having only light industry [2].

It is not only the concentration of particulate contamination that is important; it is their nature as well. Since from where I sit I can see Hebden Valley, the tragedy of Hebden Bridge comes readily to mind. In Acre Mill they wove asbestos with inadequate safeguards and much of the population, even those who had never worked at the mill, contracted asbestosis. There are all too many similar cases.

The problems facing inspectors include identifying the source of particulate matter, and determining whether it is liable to be injurious to health and whether it constitutes a nuisance. Physical and chemical assay may be required to resolve these problems.

Airborne dust in quarries, factories and mines has been causing increasing concern in recent years [257]. This has led to the design of a number of dust-sampling instruments which are now commercially available. Airborne dust is usually measured in terms of number or mass concentration. Historically, for a number count, the particles were collected in such a way that a microscope count of them could be made. This has been made easier with the introduction of automatic microscopy; the introduction of sensing zone (e.g. light-scattering) instruments has, in many cases, rendered the method obsolete. In 1959, the Johannesburg International Conference on Pneumoconiosis [3] recommended that the mass concentration of respirable dust was the best single descriptive parameter to measure in order to assess the hazard of pneumoconiosis from coal dust; for quartz dust the surface area of the respirable dust was thought to be the best parameter to measure.

As a result of these recommendations, a whole range of gravimetric dust-sampling instruments has been developed for use in coal mines, quarries and other industries with an airborne dust problem. There has been little work on surface area dust-sampling equipment although some work has been carried out on light-scattering techniques [4, 5].

Anyone living and working in a dusty environment inhales dust with the air he breathes [209]. Most of the dust is exhaled again but some is retained and can cause lung disease. The walls of the respiratory passages are lined with mucus membrane which is covered with hair-like cilia that keep up a constant ordered rhythmical movement with the force directed towards the mouth and nose. Since the factor controlling deposition is sedimentation, it is unlikely that any unit density particles larger than about a hundred μm will penetrate beyond this region. The total capacity of the lung is about 5500 cm³ but, even during activity, only about 2500 cm³ of air are breathed in and out. This is called 'tidal air' and the exchange of oxygen, carbon dioxide and particulates with the residual air is by diffusion. Dust particles smaller than about one μm diffuse very slowly and only a few per cent of these particles are deposited since they penetrate only a short distance into the residual air region [6, 8]. Davies [9] shows that only particles within the size range 0.25 to 10 μm (called respirable dust) enter the lung and only a percentage, which varies with the size of the particle and the person's retention characteristics, are retained.

In gravimetric sampling, the weight of the dust less than 0.25 μm in size is usually negligible compared with the total weight of respirable dust, hence there is no need to prevent this very fine dust from being collected. However the presence of a very few particles larger than 10 μm in size would be significant, hence a size selector, which prevents the collection of such dust, is necessary in all equipment which assess dust as a health hazard [10, 12, 215].

Irregular particles, coarser than 10 μm, are often found in the lung and this is due to their low sedimentation velocity. Asbestos can readily break into bundles of fibres having a diameter of only 0.02 to 0.12 μm but a length of 1 to 150 μm. Amosite and crocidolite have straight fibres and are more likely to penetrate the respiratory tract than chrysotile which has curved fibres. Elmes [13] considers that this is why the last-named is less likely to cause lung damage than the others.
The recommended maximum threshold values in the UK are [256]:

- chrysotile, amosite: 2 fibres cm\(^{-3}\) (0.10 mg m\(^{-3}\))
- crocidolite: 0.2 fibre cm\(^{-3}\) (0.01 mg m\(^{-3}\))
- gypsum, limestone: 10 mg m\(^{-3}\) total dust
- quartz: 10 mg m\(^{-3}\)/(per cent respirable + 2)
- coal dust: 8 mg m\(^{-3}\) respirable

Although the incidence of asbestosis can be regulated at these levels, inhalation of a single fibre of crocidolite can cause cancer.

The only statutory regulations are for coal dust (1975) and at this level a 1% level of pneumoconiosis is incurred. No compensation is available to coal miners who contract bronchial disease since it is not accepted that this is caused by working in a dusty environment. The Federal Coal Mine Health and Safety Act of 1969 defines an upper level of 2 mg m\(^{-3}\) for the States as compared with the British level of 8 mg m\(^{-3}\).

Urban industrialization and increased use of motor vehicles have led to an increasing amount of toxic material in the atmosphere. The consequent hazards and need for effective monitoring are well documented [14-16].

There is also some evidence that non-respirable dust may be a factor in the development of bronchitis [17] and in a recent survey at Hamilton, Ontario, a strong relationship was found between the atmospheric pollution index and hospital admissions for respirable diseases [18].

Fine tobacco smoke cannot be deposited to cause bronchial carcinoma but if the particles are inhaled and held in the lungs for a few seconds they can, by absorbing moisture, increase in size to about 3 μm and be readily deposited [9].

Cans should be taken that the sample accepted by any instrument is representative of the environment and that, where samples are taken from a moving airstream, the sampling should be isokinetic (Chapter 2). For non-isokinetic sampling, at a wind velocity of 4 m s\(^{-1}\), the collecting efficiency has been found to be less than 15% for particles coarser than 10 μm. External shields and covers were found to have a large effect on efficiency [19]. Efficiency may also be impaired due to re-entrainment [206].

Davies [26] gave the criteria for accurate sampling in still air:

\[
10 \left( \frac{Q}{4\pi} \right)^{\frac{1}{3}} \leq D \leq \frac{2}{5} \left( \frac{Q}{mgT} \right)^{\frac{1}{3}}
\]

where \( Q \) is the suction flow, \( T \) the relaxation time and \( D \) the probe diameter. The first term expresses error due to particle inertia and the second sedimentation error (<4%) which is not relevant when the nozzle is horizontal. The first term reduces to

\[
Stk < 0.016 \frac{D^2 \nu}{F}
\]

which is the criterion for an inertial sampling error of less than 1%. The corresponding criteria for 5% and 10% error give the numerical constant as 0.085 and 0.179 respectively.

\[ Stk = \frac{\rho_p d^2 \nu}{18 \eta D} \]

where \( \rho_p \) is the particle density, \( d \) the particle size, \( \eta \) the gas viscosity and \( \nu \) the wind velocity.

Gibson and Ogden [276] state that particles smaller than 40 μm are collected efficiently in calm air by sharp-edged orifices of diameters 3 to 10 mm aspirated at 5 to 20 1 min\(^{-1}\) pointing upwards or horizontally. Errors arise due to sedimentation for flow rates less than 5 1 min\(^{-1}\), in agreement with Davies. They state that the extrapolation by Davies of an empirical equation by Belayev and Levin is unjustified.

The effect of probe bluntness on sampling particulates from air was investigated by Sansone and Christensen [278] who found that increasing bluntness produced a coarser distribution at a sampling velocity of 40 cm s\(^{-1}\). They found no effect for sampling velocities of 20 and 4 cm s\(^{-1}\). Bluntness was defined as \((D/d)^2 - 1\) where \( D \) and \( d \) are the outside and inside probe diameter respectively.

In order to obtain meaningful data two types of instrument have been designed. Fixed-point samplers are designed to sample dust in the body of the air; the results can only be significant if the site selected is representative.

In British coal mines such instruments are sited 70 m down the return airway from the coal face [20] but they are supplemented by personal dust-sampling instruments. These latter instruments are usually carried for the whole of the working day and must therefore be small and light. Further, they should have an inlet near to the worker's nose and, as far as possible, simulate the person's breathing.

Fixed-point samplers are frequently used in controlled environments. These environments are essential in many areas of modern precision manufacturing technology where the presence of particulates can affect the reliability and performance of the product. This is especially true in aerospace equipment, electrical components and integrated circuits. Dust can also affect quality in the pharmaceutical and food-processing industries. Excessive matter can cause defects in photographic emulsions. Air contamination monitoring is also essential in hospital operating theatres.

In America the EPA specify a high-volume sampling method [293] while the Occupational Safety and Health Administration (OSHA) personnel use personal monitoring to determine particulate levels in a plant.

Duvall and Bourke [21] compared a gravimetric personal sampler having a thousandth of the flow rate of the high-volume samplers and found good correlation with EPA regulations for a 'clean' laboratory area, outside ambient air and an industrial location.

Particle size determination of collected dust is also necessary for determining the grade efficiency curves for dust-arresting plant. The techniques involved here are usually quite different to those used for sizing discharge from chimneys and flue gases, since the quantities of collected material are usually much greater and the individual particles are more robust.

Methods of sampling and measuring the size distribution of aerosols have been studied
extensively [22-27,188] and detailed surveys of equipment available have been presented [28,29]. The broad objective is to obtain a representative sample, either in such a way that the health or nuisance hazard may be estimated immediately or in such a way that it may be examined further without altering the characteristics of interest. For these reasons some sampling devices have been developed which automatically size-grade the collected particles and others which collect the particulate matter so that further examination may be carried out with the minimum of effort, e.g. gravimetric assay for concentration determination and microscopic analysis for size grading.

Many aerosol particles consist of flocs or aggregates and in collecting and redispersing them the identity of the original distribution is often lost. This may be of little importance if the toxic effect is being examined, but of fundamental importance when settling behaviour is being studied. The statistical accuracy of the collected samples is usually very low since small samples are collected from enormous inhomogeneous volumes of gases. For this reason the expense of sophisticated collecting devices cannot always be justified, and collecting devices which are expensive in terms of highly qualified operators may be similarly discarded. It is also found that particular industries have developed their own equipment. For these and other reasons a wide range of sampling devices is available. They tend, however, to fall into one of the following categories:

1. Inertial techniques, impingement, impaction and sedimentation.
2. Filtration.
3. Electrostatic precipitation.
4. Thermal precipitation.
5. Light scattering.

### 3.2 Inertial techniques

Simple sedimentation techniques are suitable for high particle concentrations. With low concentrations a deep cell is required to give a sufficiently dense deposit, but the time of sampling becomes impractically long. Sedimentation cells have the useful property that the particles are not altered physically in the course of collection; usually there is no size selection [30,31]. Particles much smaller than 1 μm in diameter are too small to be collected efficiently by sedimentation techniques. Aitken developed a method of increasing the size of particles by condensing water vapour on them thus enhancing the rate of sedimentation. The Aitken Nucleus Counter is still occasionally used [238].

Size distribution measurements may be made using the Casella settlement dust counter, in which a volume of air is enclosed in a cylinder at the base of which is an arrangement for exposing a number of microscope slides in sequence. By timing the exposures and counting the number of particles collected on each slide, a particle size distribution may be obtained.

Hallworth and Hamilton [207] describe the use of a settling drum followed by microscope analysis of slides with a Quantimet 720 for sizing of pressurized suspension.

The earliest methods of sampling gas-borne particles have incorporated impingement devices [32-34]. The principle is shown in figure 3.1. The large jet with low velocity

Sampling and sizing from the atmosphere will deposit the larger particles on the slide, while the small jet will deposit the smaller particles on a subsequent slide.

![Impingement device for collecting airborne particles.](image)

**Fig. 3.1** Impingement device for collecting airborne particles.

All impactors are designed to separate particles from gas streams by their inertia which causes them to impact against a collecting plate when the gas streams are made to abruptly change direction. Owens [35] and Ferry [36] used single jets and a dry glass slide to collect the samples which were then analysed by microscopy. A comprehensive review of early techniques used for air-pollution studies is to be found in [35].

Modern impactors both sample and classify in order to reduce the subsequent labour of the microscope examination. The most versatile and popular aerosol sampler and grader is the 'cascade impactor' developed by May [37], and many variations of the instrument exist (figure 3.2). The May cascade impactor samples liquid or solid particles from 0.5 to 50 μm diameter and deposits them in four size fractions on separate glass discs. The intake velocity is kept deliberately low in order that delicate particles may be deposited without damage; for this reason it is particularly useful for establishing the efficacy of sprays for spreading insecticides, fine mists, atomized liquids, as well as pollen, spores, smokes and other delicate structures. This use of low velocities has been criticized on the grounds that high Reynolds numbers are required to give sharp cut-off characteristics [38].

The glass collecting discs are coated with a medium suitable for the material to be collected. For solid particles, a non-drying, sticky film is used. For liquid particles, dyes etc., other media have been devised. The coated discs are loaded into each of the four stages; a filter paper, if required, is put into the fifth stage for the collection of fines and a source of suction is connected to the outlet pipe. After running for a given time, the discs are unloaded and mounted on glass slides. The slides are evaluated by strip counting or, for more accurate analysis, a Porton graticule (see Chapter 6) is used in the microscope eyepiece. A modified impactor extending into the sub-micron range was described by Sonkin [244].
The impaction process was examined theoretically by May and others [39, 40], all of whom showed that the impaction efficiency is related to a dimensionless parameter $\psi$ given by:

$$\psi = \frac{d^2}{\eta W} \times \text{constant} \quad (3.2)$$

(In a later paper May [41] quotes the constant as $C/18$ where $C$ is Cunningham's slip correction factor.) $\rho$ is the material density, $v$ is the gas jet velocity at the orifice, $d$ is the particle's aerodynamic diameter, $\eta$ is the air viscosity and $W$ is the diameter for a circular jet or the width for a long slit.

May's impactor has been used by Laskin [42] on heavy aerosol particles. Other writers have carried out extensive theoretical and experimental work with cascade impactors [43, 44]. Characteristics and designs of impactors have also been improved [45 - 47]. Special duty impactors have also been developed, e.g. Brink's five-stage cascade impactor for sampling and grading acid mists in the size range 0.3 to 3.0 $\mu$m [48]. Davies and Aylward [39] in a theoretical study of rectangular jets, showed that the sharpness of the cut should improve if the jet-to-slide clearance is reduced to approximately three-eights of the jet width.

The Unico cascade impactor is designed to operate between 2 and 40 $\text{mln}^{-1}$ and uses standard microscope slides for its five impaction surfaces. A special feature is the manual slide-moving mechanism which allows the slides to be moved in four equal increments up to the total, permitting the collection of nine times more sample than is possible on a non-moving slide.

The Casella cascade impactor samples at the rate of 17.5 $\text{mln}^{-1}$, on four 1-in diameter glass discs. Soole [56] tested it, with particular reference to its calibration. Impactor calibration was discussed by Berner [50] and data handling and computer evaluation were described, for the Casella counter, by Picknet [51].

Stevens and Stephenson [258] describe a single-stage size-selective dust sampler in which the particle size deposition curve is nearly identical to that for the pulmonary compartment of the lung as proposed by the ICRP Task Group on Lung Dynamics (1966). Casella market this instrument as a personal sampler operating at 21 $\text{mln}^{-1}$.

Stevens and Churchill [259] later describe a high-flow-rate version (35 $\text{mln}^{-1}$) which can be operated as an environmental sampler. This instrument is manufactured by Bird and Tole Ltd.

Buchholz [52, 53] describes a low-pressure cascade impactor and Bürkholz [54] uses one for droplet size determination in the 0.5 to 10 $\mu$m range. Jacobson et al. [55] describe a new nidget impactor.

The Greenburg-Smith impinger [32] employs a jet and plate immersed in a liquid of low volatility so that the arrested particles are retained in suspension in a liquid [57 - 64]. ASTM Standard D 3365-74T describes the use of this instrument in conjunction with a Coulter Counter (see Chapter 13).

Kotrappa [65] discusses the determination of mass median diameters using particle shape factors. Pelassy [66] calculated the theoretical efficiency and deposition pattern for a Casella Mark 2 impactor. Marple [67] considered the fluid mechanics of the laminar flow impactor and compared theory and practice for round and rectangular jets. Hänel [68] considered jet impactor characteristics versus relative humidity and found that the cut-off size increased with increasing humidity. In 1975, May [41] examined the possibility of preconcentrating particles down the axis of an air jet in order to increase efficiency. Other parameters examined were the origin, magnitude and elimination of halo deposits; the effect of nozzle shape on impaction performance and the validity of the characteristic impactor parameter. He also introduced an 'ultimate' cascade impactor [70]. From the three alternatives available to him he preferred slots to single or multiple circular jets.

Thomas [71] preferred the horizontal elutriator to the cascade impactors using several, of different capacities, in parallel. They have the advantage, over cascade impactors, in that the cut-off size can be determined theoretically from the efficiency equation by putting $E$ equal to unity:

$$E = \frac{b L n v_p}{q_t} \quad (3.3)$$

where $E$ is the efficiency, $b$ the slit width, $L$ the slit length, $v_p$ the particle velocity and $q_t$ is the volume flow rate through $n$ channels.
The relative merits of dry and coated impaction surfaces were investigated by Dzubay et al. [72], who found that dry surfaces of aluminium foil, glass-fibre material and wax-coated paper gave distorted particle size distributions when compared with grease-coated surfaces, where particles bounced off the dry surfaces and were collected at a later stage.

Rao and Whitby [271] found that the retention efficiency of a single-stage impactor to polystyrene latex in the size range 0.79 to 1.1 μm rises from 0 to 100% for oil-coated plates; for uncoated plates the rise is from 0 to 50% followed by a fall. They also found that glass-fibre filter collectors minimize bounce but modify collection characteristics [272] and that some Whatman filters are unsuitable as collection surfaces.

Conventional impactors utilize a solid treated or untreated surface from which particles rebound, re-entrain and agglomerate leading to erroneous results. In ‘virtual impactors’ the solid surface is replaced with a slowly pumped stagnant air void [273–275].

Marple and Rubow [73] designed an impactor specifically for calibrating optical counters. A single calibration point is obtained by connecting the impactor on to the inlet of the optical counter and comparing the particle size distribution with that obtained without the impactor. Various calibration points were obtained by using different diameter nozzles. The possibility of using two impactors in series was also investigated but found superfluous for calibration purposes.

The Brink Model B Cascade Impactor can be used under ambient conditions or at elevated temperatures and pressures encountered in process gas streams. A glass cyclone is used upstream of the impactor to remove all particles greater than 7 μm. The impactor then collects the rest in five steps down to 0.3 μm.

In the Konimeter [74] a measured volume of air is sucked through a narrow aperture at high velocity by means of a spring-loaded piston. The particles impinge on a slide coated with adhesive, so that the particles may be counted microscopically. The collecting efficiency of this instrument is reported to be low, aggregation on the slide makes counting difficult, and the instrument cannot be recommended for size analysis [75]. Sizing may be speeded up with this instrument using a newly developed scanner [75].

The Sartorius Konimeter is recommended for routine and control measurements in factories where dust containing quartz, cristobalite, tridymite, asbestos etc., are present. Quantitative separation of particles in the size range 0.7 to 5 μm is effected and a built-in microscope permits fast evaluation of the dust hazard.

In the Owens jet counter [35] and the Bausch and Lomb dust counter [76] the air passes through a humidifying chamber and then expands through a narrow slit. Moisture condenses on the particles, thus aiding the deposition. Such devices have a very variable efficiency depending on the type of aerosol being examined [75].

Cunningham et al. [77] report on a programme for classifying airborne particulate material with respect to size and type using inertial impaction. The major chemical constituents were measured using infrared spectroscopy and showed variations of the chemistry with size and time. This programme was initiated as a result of the Pasadena Smog Aerosol Study [78] and followed a similar study by Whitby et al. [79–81], who concluded that in a photochemical smog sub-micrometre particles arise from condensation and coagulation of photochemical reaction products, whereas larger particles arise from reevaporation or other mechanical sources. Cunningham used a four-stage Lundgren impactor [82, 83] which uses rotating drums as the impaction surface in each stage. The impactor operates at a flow rate of 6.8 m³ hr⁻¹ and collects samples with mass-mean diameters of 1.2, 1.2 and 0.3 μm. Using a continuous drive mechanism, it was operated for 21 continuous hours and later, using a stepwise mode of operation, for 3 days with 3 hours at each step. The particles were deposited on a Mylar film and the size distribution determined by weighing. The Lundgren impactor has also been used in comparison studies with a light-scattering counter [84] and to determine variation of particle size concentration and composition with time [85].

Leary [86] has used an autoradiographic technique in which radioactive particles are sized through their emission. A filter carrying a deposit of radioactive particles is placed in contact with a photographic plate for selected exposure times. The number of tracks is counted and an equation applied to calculate the size of each particle. The technique can only be applied to small particles (0.1 to 20 μm) and has the advantage that agglomerates may be distinguished from single particles. Cowan [87] has reported on a simpler method which depends on the formation of a visible spot on the film.

In the Cascade Centrifuge [88–90] air is sampled at 30 l min⁻¹. When the air stream passes through an orifice, the central flow lines pass right through, and the peripheral flow lines are deflected sharply and particles separated from them by their inertia. A series of three orifices of diminishing diameter are used to give cuts at 14, 4 and 1.2 μm.

This has been used, together with a Casella impactor and a multi-stage liquid impinger to analyse inhalation aerosols [208].

In order to determine the concentration of respirable dust in a cloud it is necessary to remove large particles which do not reach the lung and efficiently collect the fines. In a study of collection through vertical and horizontal ducts (elutriator), Walton [91] showed that, with the former, the sampling efficiency is 1 – (f/v), where the sampling efficiency is defined as the fractional number of particles falling speed v collected with an intake velocity v; with the latter, the sampling efficiency for a volume flow rate Q is 1 – A/Q, where A is the horizontally projected flow area of the duct. As a result of these studies, a horizontal elutriator was developed with a cut at a falling speed of 0.15 cm s⁻¹ which is equivalent to a 7 μm sphere of density 1 g cm⁻³ falling in air; later instruments were constructed to give a cut at 5 μm [137]. The sample is collected on filter paper and may be examined gravimetrically or by using a microscope. In the discussion following Walton’s paper Dawes drew attention to field work carried out by the Safety in Mines Research Establishment (SMRE) with a horizontal elutriator [92] in which it was found that the size selection characteristic was not stable with time, but that over an eight-hour period the cut size rose from 7 to 10 μm due, it was suggested, to redispersal of dust collected on the elutriator plates caused by the entrance of large particles. This effect was not noted by Wright of the Pneumoconiosis Research Unit of the Medical Research Council in Cardiff, who was working on an elutriator now available as the Hexlet, which was similar to that of Walton. In this instrument, unit density spheres larger than 7 μm diameter are collected in a Soxhlet thimble and the weight is
determined gravimetrically. The aspiration rate is 50 l min\(^{-1}\) compared to the 2.5 l min\(^{-1}\) of the gravimetric dust sampler [247].

Ashford and Jones [248] compared the Hexlet and Conicycle airborne dust samplers and standard and long-running thermal precipitators at three collieries and concluded that the Hexlet was unsuitable for prolonged underground work.

The SMRE work resulted in the development of several instruments for sampling respirable dust in coal mines and these are marketed by Casella. The Simpald [93 - 95] is based on a parallel plate being collected on a membrane filter. Suction is effected by ejecting a stream of carbon dioxide gas from a small nozzle into the diffuser throat.

In the Simpald [96, 97], the parallel plate elutriator is replaced by cyclones. The instrument weighs only 805 g and samples at the rate of 1.85 l min\(^{-1}\), taking its power from the miner’s cap lamp battery.

The BCIRA [246] gravimetric size-selecting personal dust sampler and the Unico respirable dust sampler which consist of a cyclone and a filter, are used by the Factory Inspectorate [29] in the manner described by Crosby and Hamer [292].

Lippmann [249] discusses the relative merits of elutriators and cyclones for removing the non-respirable dust fraction and prefers the latter.

A multi-stage impactor was described by Andersen [98] for use primarily with biological material in which the gas stream passes through small circular holes in a plate and impinges on a prepared surface in a Petri dish. A calibration of this impactor has been reported [99], and a simplified application to the study of airborne fungus [100].

A later model was designed for respiratory health hazard assessment [237].

Andersen Inc. make a range of these multi-orifice samplers including one small enough for in-stack sampling (see Chapter 2). The HI-Vol impactor designs permit fractionation in five, three or two sizes ranges from 7 down to 0.1. This instrument has been tested at an EPA site and subsequently adopted for use in their Community Health Effects Surveillance System (CHESS) sampling stations [101]. A procedure of changing filters to minimize losses has also been devised [102], a description of calibration procedures published [103] and its use in characterizing suspended airborne particulates presented [104, 269]. Andersen also manufacture an ‘Ambient Sampler’ for in-plant use together with a ‘Mini Sampler’, all based on the multi-orifice principle.

Rao and Whitby [272] describe the calibration of a four-stage Lundgren and a six-stage Andersen with oil particles. For the former, calibration is in good agreement with theory; for the latter, the calibration curves are less steep and are shifted to a higher aerodynamic diameter. The peak efficiency is very sensitive to the jet used and to some extent particle size.

May [261] modified the Andersen sampler to have a single jet and inlet tube at the topmost stage. This was done to improve the large-particle performance over the known poor efficiency of the standard multiple-hole arrangement. May [260] also suggested that the efficiency would be improved by replacing the conventional 400 holes by 200 holes of 1.85 mm diameter arranged in a radial pattern.

A cascade impactor was designed by Newton et al. [277] according to basic principles. By defining flow characteristics at each stage, within defined ranges, the collection efficiency of each stage remained sharp throughout the impactor and effective cut-off diameters were in accord with those predicted. Another advantage is a high-flow-rate impactor. Suggested criteria were: 1<s<2 : 1<T<5 : 500<N<3000 : for round hole jets, where s = jet-to-plate separation; w = jet diameter; T = jet-throat length.

P.L. Anderson [69] described a small centrifugal aerosol collector with a design that included multi-orifice cascade impactor plates. Spectral calibration for the size range 3.0 to 0.16 µm is also included. The centrifuge is thermally protected and small enough for in-stack applications. The particles are collected individually for electron microscopy and micromorphological techniques.

Other commercially available equipment include the Heming Aerosol Spectrometer which samples airborne particles, classifies them according to their Stokes velocity and deposits them on glass slides for later examination. The instrument is intended for long-term sampling and is available for use in coal mines.

The Rotorod [105] consists of a 20-cm length of 0.159-cm square section brass rod bent to form a C-shape with two vertical arms 6 cm long, 8 cm apart. The centre of the shaft and level part is fitted to the shaft of a 6 - 12 V, 2500-rpm electric motor. The two vertical arms sweep out 120 l min\(^{-1}\) air and collect particles by impact on the leading edge. The Rotorod is simple and portable with a tiny power requirement, high sampling rate, indifference to wind speed and direction and turbulence. It has the disadvantages that the collecting surface is easily overloaded and awkward to handle for later microscope examination. It is of little use at sizes smaller than 7 µm at which size the collection efficiency is 50% [106].

The Hirst spore trap [107] is for long-period sampling of airborne spores and pollen. The Edwards sampler [108] consists of a holder for a 1.8 cm diameter filter mounted on a servo-operated wind vane to keep the filter facing upwind. The Tilting impinger [109], designed for high recovery of large airborne particles, consists of a cylindrical flask, 2 cm in diameter and 5 cm long, with a 6.5-mm hole near the bottom through which air is aspirated.

May [110] carried out an investigation of sixteen sampling techniques for windborne particles: (1) The Rotorod, (2) Hirst spore trap, (3) Casella Impactor Mark I, (4) Casella Impactor Mark II with a guard tube, (5) Cyclone (to dimensions of Decker et al. [111] (6) Cyclone (as above with smaller (1 cm) diameter nozzle), (7) Edwards sampler, (8) Tilting impactor, (9) Ideal sampler [109], (10) wind-operated impactor [112], (11) glass ribbon on a wind vane, (12) multi-stage all-glass liquid impaction [113], (13) MDC sampler (modified Andersen cascade impactor), (14) modified Andersen lying horizontal, (15) modified Andersen lying vertical and (16) Pagoda [260].

May concluded that the Casella Impactor Mark II with a guard tube was the best instrument, having a high efficiency of down to 1 µm. In the absence of electric power May recommended the use of the Rotorod. The Hirst spore trap had a low and inconsistent efficiency and was, in general, disappointing. The glass ribbon on a wind vane was the simplest and the Tilting impactor was the only reasonably satisfactory low-sampling-rate instrument. The Andersen cannot be recommended for large-particle sampling.

The Confuge, which consists of a cone and shell rotating at 3000 rev min\(^{-1}\), draws in 25 ml min\(^{-1}\) dusty air (see figure 3.3). The indraw cloud is minnowed by an intern
Sampling and sizing from the atmosphere

As a result of their deliberations Stöber developed a centrifugal version of the gravitational horizontal duct separator [125]. In the spiral centrifuge aerosol flow rates of 3 l min⁻¹ can be fed into the rotor at the centre of rotation where it is entrained in a laminar flow of clean air. Best results are obtained at a speed of 3000 rpm to give a deposit, in the size range 0.08 to 5 μm, on a 180-cm long strip foil which forms the outer wall of a spiral duct cut into a plane disc mounted on the centrifuge rotor.

The calibration procedure was outlined in a later paper [126] in which it is stated that a number or a weight distribution was determinable. Forstendorfer [127] applied the instrument using radioactive markers, and later compared the results [128] with a laser spectrometer LASS [129] finding reasonable agreement, with the centrifuge results a little low. Kops [130] found that a correction factor had to be applied in order to obtain reliable results. Oeseburg [131] determined the particle size distribution of diocyl phthalate aerosol and found good agreement with High Order Tyndall Spectra (HOTS). An examination of the spiral centrifuge revealed that the deposition of particles in the inlet was higher than previously reported and that the experimental results were not compatible with theory [124].

Berner and Reichelt [132, 133] introduced a new concept in their cylindrical aerosol spectrometer which greatly simplified the construction and operation. They allowed the complete inlet system to rotate with the spectrometer thus eliminating the need for rotating seals. When the spectrometer is rotated, a pressure drop across the inlet to outlet nozzles draws in air, the flow rate being controlled by nozzle size and speed of rotation. For their ‘clean’ air, however, they used room air which restricts the use of the instrument [134]. To overcome this limitation Hochrainer and Brown [135] designed two centrifugal spectrometers, one cylindrical and one conical in design, having an inlet system similar to Berner and Reichelt’s as well as a method for furnishing clean air.

This design was further modified by Matteson and Boscore [136] who designed a cylindrical-type spectrometer into which aerosol particles entered a horizontal centrifugal field through two ports, a pin-hole orifice opposite a larger port. The aerosol through the larger port is removed in a cumulative size deposition on half a foil and serves as clean air for the aerosol through the other port which is deposited in a thin streak.

In 1975 Oeseburg and Roos [270] improved the bearing system of the Stöber aero spectrometer by the introduction of bearing rings to reduce leakage.

3.3 Filtration

Most filtration methods are unsuitable for collecting solid aerosol particles for microscopic analysis, since aggregation tends to occur on the filter if concentrated aerosols are sampled, while smaller particles tend to precipitate deeply between the fibres of the usual fibrous material used as filtering media so that they cannot be microscopically counted.

Filtration is the simplest method of removing particles from the atmosphere for subsequent analysis; however, removal of particles from a filter and dispersion prior to si
analysis will usually completely alter the size characteristics of the sample due to the breakdown of flocs. The most efficient filter media for this purpose are the membrane filters which can exhibit almost 100% collection efficiency for particle size above 0.01 μm with the bulk of the material deposited on or near the surface. These filters consist of cellulose esters which are soluble in acetone, so that it is possible to dissolve away the filter and transfer the deposited material to prepared surfaces for further examination [Kalms [138]]. The deposited particles may be examined in situ, using reflection microscopy, or the membrane can be made transparent by adding a few drops of cedar oil and examination carried out by transmission microscopy.

Although filtration, followed by sizing, is tedious and inaccurate, the method is extremely useful for determining mass concentration. This is usually carried out gravimetrically although other methods are also employed. Kroes et al. [139] for example used adsorption to obtain a sensitivity of 8 μg cm⁻² on their tellur filter which they recommend as superior to the Millipore-type filter. Luke et al. [210] collect particles on a Millipore filter and ash it; the amount of metallic particulates is then determined by X-ray spectrometry.

Handling of filters can present problems and to overcome these many manufacturers use a cassette filter system. Instead of changing only the filter, the whole cassette is removed for subsequent analysis under laboratory conditions.

Rotheroe and Mitchell market a wide range of dust samplers which incorporate Millipore membrane filters. The L30, L60, L90 are suitable for continuous operation at flow rates of 30, 60 and 90 l min⁻¹ respectively. The L2C is a pocket-sized personal air sampler (weight 1.14 kg, flow rate 0.5 to 3.0 l min⁻¹) to sample air close to the worker’s face.

C.F. Casella also use Millipore membrane filters in their range of instruments whereas Sartorius use their own membrane filters. The Sartorius Dust Sampler EM100 is a high-performance dust sampler with continuously adjustable air throughputs of up to 100 m³ hr⁻¹ and a filtration area of up to 415 cm². Isokinetic sampling in closed aerosol systems and fluxes is possible with instant read-out of momentum flow rate, total flow and gas temperature, so that volumes can be converted to standard m³. Applications include efficiency tests on cyclones and electrostatic filters, checking of dust concentration in exhaust flues, immision monitoring and supervision of spray-drying plants.

The Sartorius Dust Sampler Gravicon is a dust-collecting instrument for taking dust samples for gravimetric, chemical and mineralogical analyses. The Porticon is a small, portable battery-operated instrument. The air intake velocity corresponds to that of inhalation through the nose. Dust samples, taken at work stations, therefore show a particle size distribution relative to that in the respiratory tract.

The Sartorius Dust Sampler Gravicon VC25 classifies dust into coarse (>5 μm) and fine (<5 μm) and collects the two samples separately on a filter which can be evaluated to monitor dust conditions at individual working sites.

The Heming air pollution monitor monitors atmospheric pollution by sucking the air through a moving filter paper at a rate of 42 cm³ s⁻¹ using a diaphragm vacuum pump. The pressure drop across the filter increases with deposition and is a measure of pollution level.

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The Frieske and Hoepfner type FH62A dust monitor samples air at fixed times and the particulate matter is deposited on a filter tape and the mass deposited determined β-ray adsorption from a krypton-85 source of activity 50 mCi.

3.4 Electrostatic precipitation

When a charged particle passes between two electrodes carrying a high electric potential, it will move normally to the direction of flow under the force of the electric field.

The electrostatic precipitator, based on this principle, consists of an ionizing cathode at a high potential surrounded by a collecting anode; typically, these consist of concentric cylinders, the inner one often being a single wire. The dust passes between the cylinders, picks up a charge and travels to the anode where the charge is deposited. For extremely small particles, the charge consists of an excess or deficiency of one electron and the transfer of electrons from one electrode to the other constitutes an electric current which may be measured with a micro-ammeter. The magnitude of the current indicates the number of particles and their size may be found by varying the flow rate or the applied potential [140 - 142]. Details of the design of an ion chamber are given by Hurd and Mullins [143]. This particular equipment has two flat circular plates parallel to each other and maintained at a fixed potential difference.

The trajectories of particles in an electrostatic field depend on their size and their electron charge. Since particles larger than about 0.01 μm may have an excess or deficiency of more than one electron, more than one size of particle may be deposited at the same point. An instrument that uses this technique with some success has been developed by Yoshikawa [144]. In order that a microscope count may be carried out the particles are collected on membrane filters.

Podol’ski [145] describes an instrument based on classification in an electrostatic field by differences in charges connected explicitly to particle size. Seven fractions are produced in the size range 0.4 to 40 μm with a reproducibility better than 3% and an analytical time of 3 min.

Instruments based on this principle have been used for aerosols of bacteria [146, 147], and an improved instrument has been described by Morris et al. [148]. This instrument consists of a cylindrical glass tube 12 in long and of 2 in diameter with a central electrode. The inner surface of the cylinder is coated with a suitable material to act as the other electrode and to collect the samples. With the central electrode at 10 kV and a flow rate of 10 to 20 1 min⁻¹, the collection efficiency was found to be 100%. In one version the cylinder is rotated and contains up to 10 ml of liquid, so that collection in liquid could be effected directly. The principal advantages of this type of instrument are high collection efficiency over a wide size range, low resistance and high flow-rate capacity [149]. It is not very suitable for number concentration measurements. Microscope analysis of the deposited particles may be simplified by placing electron microscope grids [150] or transparent plastic over the anode. Several other studies of this technique have been carried out (see [151, 152]). Byers et al. [211] use a Bendix Mode 959 electrostatic precipitator to collect samples which they subsequently analyse by
scanning electron microscope using a computerized technique to determine projected area.

In 1938 Barnes and Penney [240] described a dc precipitator capable of efficient sample collection at a flow rate of 3 ft³ min⁻¹. Commercial versions are available from Bendix and Mines Safety Appliances.

Two commercial versions of high-volume electrostatic precipitators described by Decker et al. [111] are available as the LEAP and the Litton high-volume (10 000 ft³ min⁻¹) samplers. Other instruments are available from Gardner Associates (the electrostatic bacterial air sampler) and Del Electronics (the high-volume electrostatic precipitator). Collection efficiency determinations of the latter have been reported [242].

![Diagram of the Thermo-Systems Model 3100 Electrostatic Aerosol Sampler.](image)

The Thermo-Systems 3100 Electrostatic Aerosol Sampler (figure 3.4) consists of a charging section and a collecting section. A vacuum pump draws the aerosol through the system at a constant rate. As the aerosol passes through the charging section, the particles are subjected to alternating pulses of positive ions generated by a corona discharge from a fine wire. The positively charged particles then flow through the collecting section. A positive voltage periodically applied to the upper plate drives the particles to the lower surface. After sufficient time to deposit all charged particles, the voltage on the upper plate is shut off. The continuous aerosol flow through the chamber again fills it with charged particles. The unique separation of the charging section and the collecting section, together with the pulsed precipitating voltage produces a uniform, representative sample, which can be evaluated without bias due to particle characteristics. The instrument allows precise determination of the sampled aerosol quantity because this volume is completely independent of the flow rate through the instrument.

Volume depends only on the number of precipitating pulses or cycles and the volume directly above the sample surface. These characteristics are in marked contrast to normal electrostatic samplers. A second mode of operation facilitates the collection of a sample when classification due to particle characteristics is tolerable. In this mode, a constant dc precipitation voltage collects all charged particles soon after they enter the collection section. The size range is 0.02 to 10 μm at a flow rate of 4 to 10 l min⁻¹ (6.7 to 167 cm³ s⁻¹).

The point-to-point electrostatic precipitator [243] was designed to collect aerosol samples for particle size analysis by electron microscopy. Its function is therefore different from the other instruments described above, which were designed to collect large samples for mass concentration analysis.

3.5 Electrical charging and mobility

The Electrical Aerosol Analyser was described by Whitby in 1966 [262]. Calibration was effected by capturing the particles after they had passed through the instrument a measuring them by microscopy [263], hence problems were caused due to the difference between the projected area diameter and the aerodynamic diameter. In a second method, a polydisperse aerosol was generated and passed through a differential mobility analyser, where monodisperse particles are separated from the airstream and used for calibration. A version of this instrument is manufactured by Thermo-Systems (see figure 3.5); its performance has been examined [212, 264, 265, 279] and its use, together with various other aerosol monitors, has been described in an airborne particle monitoring facility [266]. The mode of operation is as follows:

As a vacuum pump draws the aerosol through the analyser, a corona generated by a high-voltage wire within the charging section gives the sample a positive electrical charge. The charged aerosol flows concentrically from the charger to the analysing tube sector, which is an annular cylinder of aerosol surrounding a core of clean air. A metal rod, to which a variable, negative voltage can be applied, passes axially through the centre of the analyser tube. Particles smaller than a certain size (with high electrical mobility) are drawn to the collecting rod when the voltage corresponding to that size is on the rod. Larger particles pass through the analyser tube and are collected by the current-collecting filter. The electrical charges on these particles drain off through an electrometer, giving a measure of current. A step increase in rod voltage will cause particles of a discrete larger size to be collected by the rod with a resulting decrease in electrometer current. This decrease is related directly to the number of particles in the aerosol between the two discrete particle sizes. A total of 11 voltage steps divide the 0.0032 to 1.0 μm size range of the instrument into 10 equal geometrical size intervals. The 11 internal boundaries are 0.0032, 0.0056, 0.0100, 0.0178, 0.0316, 0.0562, 0.1000, 0.1780, 0.3160, 0.5620 and 1.0000 μm. Different size intervals can be programmed via an optional plug-in memory card.

The instrument can be operated either automatically or manually. In the automatic mode, the analyser steps through the entire size distribution range. For size and concentration monitoring over an extended period of time, the analyser may be intermit-
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tently triggered by an external pulse from a timer. The size distribution can therefore be automatically monitored at preset time intervals. The standard read-out consists of a digital display within the control circuit module. This module programmes the analyser voltages and also provides command logic for the automatic counting mode.

Reviews of electrical measuring devices have recently been published [213, 286] together with descriptions of their applications [216, 217].

Fig. 3.5 Thermo-Systems Model 3030 Electrical Aerosol Size Analyser.

3.6 Thermal precipitation

Dust particles in suspension move away from hotter to colder regions [153, 154]. This effect was noted first by Tyndall and later by Lord Rayleigh, and Aitkin showed that the force that caused this movement was thermal in origin.

For particles of the same order of size or greater than the mean free path of the gas molecules, an equation may be developed using ‘thermal creep’ theory. This is based on the force set up at the gas-solids interface between a particle and the surrounding gas. When the gas temperature increases along the surface, the molecules leaving the surface will have a greater component of velocity in the direction of the temperature increase than when they arrived at the surface. The net result is a creeping flow of gas from the colder to the warmer regions along the surface of the particle giving a net force in the cold direction. This motion is called thermophoresis [239].

Sampling and sizing from the atmosphere

Epstein [155] developed the following equation for the thermal force:

\[ F_t = \frac{9 \pi \eta d}{2 \rho f T} \frac{dT}{dx} \]

where \( d \) is the particle diameter; \( \eta \), the gas viscosity; \( \rho_f \), the gas density; \( T \), the gas temperature; \( dT/dx \), the thermal gradient; and \( \chi_g \) and \( \chi_p \) are the thermal conductivities of the gas and particle respectively.

Equating with Stokes resistance, including Cunningham’s correction, gives the velocity in the thermal gradient as:

\[ U_t = \frac{3 C \eta}{2 \rho f (2 + \frac{x_g}{x_p})} \frac{dT}{dx} \]

where \( C = (1 + 24 \lambda / d) \), see equation (7.18). This equation is found to give good agreement with experimental results.

For very small particles, equations based on the kinetic theory of gases have been developed [156]. Waldmann’s equation may be written:

\[ U_t = \frac{1}{(5 \pi P)} \frac{x_g}{P} \frac{dT}{dx} \]

where \( P \) is the gas pressure. For further details readers are referred to [157, p. 417].

This principle is applied in thermal precipitation where the hot body is an electrically heated wire placed between two collecting plates. The thermal precipitator developed by Watson [158] (figure 3.6) consists of a channel 0.51 mm wide between two microscope cover glasses with an axially situated wire heated electrically to about 100°C above ambient.

A dust sample, about 9 mm long and 1 mm wide, is collected on each plate. The normal flow is 7 cm² min⁻¹ and collection efficiency is high for sub-5 μm particles. Larger particles are collected elsewhere in the instrument. The collecting device may be modified so that the sample is collected directly on an electron microscope grid, although transfer of particles from the collecting plate to a suitable film and thence to a grid is a more usual procedure. This instrument is manufactured by C.F. Casella & Co., and also distributed by Mines Safety Appliances [177].

Direct measurement of collection efficiency indicates that this type of instrument collects virtually all particles from 5 to 0.01 μm in size [159–162]. Sampling efficiency has been found to be unaffected by airspeeds up to 6 m s⁻¹ [163].

Modifications to the traditional design [164] include a means of centring the wire in position [165], the substitution of a ribbon for the wire to give a more uniform deposit [166], the provision of an inlet elutriator to exclude coarse particles [167], and the attachment of a rotating magazine containing six pairs of cover glasses to avoid the con-
tamination arising when glasses have to be transferred from the sampling head to a slide box [168].

The instrument of Beadle and Kitto [169] deposits 12 dust strips on a slide and also employs an inlet elutriator and a bellows driven by clockwork. A reversible water aspirator, much smaller and more convenient than the standard form, has been developed by Wright [170] and is manufactured by Adams. Long-running thermal precipitators may be operated for periods of hours rather than minutes. In that of Camber et al. [171] the sample slide rotates continuously; in Walton's [172] the slide oscillates and in that of Orr and Martin [173] the slide is replaced by a transparent tape.

Hamilton's long-period (8 hours) dust sampler is the standard instrument for determining dust concentrations in British coal mines [174, 182] (Casella & Co.). The air enters through an inlet elutriator, then passes over a horizontal slide where particles down to 1 or 2 µm are deposited by gravity settlement; the fine particles are deposited at the far end of the slide by a hot ribbon. Aspiration at 2 cm$^3$ min$^{-1}$ is by an electric pump energized by the battery that heats the wire.

Balashov et al. [175] have used a composite instrument to obtain samples at very low concentrations. Thermal precipitators sampling large volumes of air and providing dust deposits large enough for weighing have been developed [176]. Casella market one instrument for short-period sampling in the size range up to 20 µm for evaluation of health hazards, such as pneumoconiosis and another for long-period sampling of respirable dust [177].

The Thermopositor (figure 3.7) collects samples of dust, smoke, fog, bacteria, pollen and spores. Air at up to 1 l min$^{-1}$ is drawn into the space between two discs, the upper
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one being heated and the lower one cooled. The particulate matter is deposited on a substrate of glass, paper, metal foil or membrane.

The Konisampler is a compact thermal precipitator which operates at up to 50 cm$^3$ min$^{-1}$ with a collection efficiency of 100% for sub-5 μm particles. The microthermal precipitator is designed to be fitted on the stage of a microscope so that particles may be observed during sampling. Ficklen manufactures these and three other thermal precipitators: 'Continuous', 'Oscillating' and 'Gravimetric'.

3.7 The quartz microbalance

In 1971 Olin [178 - 180] described a system in which aerosols are drawn into an electrostatic precipitator. A piezoelectric quartz oscillating crystal forms one electrode, the resonance frequency of which decreases linearly with increasing total mass of the particles thereby weighing them. Several commercial versions are now available and papers have been published on their applications [214].

With the Thermo-Systems 3200 Series Particle Mass Monitor, aerosol is drawn in at a flow rate of up to 1.01 min$^{-1}$ (17 cm$^3$ s$^{-1}$) and deposited on to a crystal oscillating at its resonant frequency. A second sensor, the reference quartz crystal, compensates for possible changes in ambient effects. The time rate of change of the output frequency signal is a measure of the total mass concentration. The sensitivity is 1 Hz, equivalent to 0.006 μg with a total loading of 50 μg. The instrument collects particles in the size range 0.01 to 10 μm approximately at concentrations between 2 to 20 000 μg m$^{-3}$.

The Ceresa Portable Particulate Mass Monitors are very similar to the above. Model PM-39 also has a small rotating disc which collects a particulate sample over a predetermined period to provide a time-correlated record. Size range is 0.1 to 100 μm.

The Berkeley/Ceresa Model C100A QCM cascade uses ten stages of quartz crystal microbalances and inertial impactor nozzles to measure the mass distribution directly in the size range 0.05 to 25 μm at concentration levels from 0.1 to 65 mg m$^{-3}$. The instrument may be coupled with a Texas programmable computer T159 and a PC-100A printer to quickly monitor and compute stage concentrations and plot particle size distributions.

3.8 Light scattering

The scattering of light by particles suspended in a fluid has been widely used in the development of instruments for sizing aerosols. The basic requirement is that particles pass through the sensing volume in single file, hence the aerosols have to be very dilute. The radial distribution of scattered intensity is a function of particle size and shape, the wavelength of the incident radiation, the refractive index of the particle and the relative refractive index between the particle and its surroundings.

The modern theory of light scattering as developed by Mie is dealt with in detail by van de Hulst [181]. Two types of instruments have evolved, the right-angle light-scattering system and the near-forward light-scattering system. The former has the advantage of being very sensitive to small changes in particle size but has the disadvantage that the scattering is strongly dependent on the particle refractive index making it unsuitable for use with mixed aerosols. Particle refractive index has little effect on the intensity of the forward-scattered light but this tends to be rather weak for particles smaller than 1.5 μm.

In an early instrument developed by Gucker et al. [182, 183] a stream of aerosol particles in a sheath of filtered air is passed through a light beam and the light scattered in a forward direction, between 1° and 20°, by each particle in transit is collected by an optical system which incorporates a photocell. The resulting electrical pulses are amplified, sized and sorted using a multi-channel pulse height selector. The sheath of filtered air plays a dual part in that it reduces the concentration of the aerosol and maintains it in a central position in the sensor and it reduces contamination to the sensor windows. In later instruments [184], right-angle scattering was used.

Right-angle scattering is used in the Aerosoloscope [185] which draws in air at the rate of 1.81 min$^{-1}$ and dilutes it to give a maximum count rate of 2000 particles per minute. The resulting electrical pulses are graded into 12 size channels corresponding to sizes in the 1 to 60 μm range at initial concentrations of up to several thousand particles per cubic centimetre.

Oeseburg [186] calculated that particle size response characteristics for a sphere of refractive index 1.491 and size parameter 0.1 < (nd/λ) < 50 are best for a forward receiver angle of 20° to 120°.

There is a boom in this type of instrument at the present time due to pending and enacted legislation. Many controlled environment operations are conducted according to USA Federal Standard 209 (1973). This classifies rooms according to the maximum allowable number of particles per unit volume with equivalent diameters equal to or greater than 0.5 and 50 μm. Another application is the inspection of 'clean benches', i.e. work surfaces continuously bathed in ultrapure air to ensure operations are carried out in a class 100 environment (no more than 100 particles greater than 0.5 μm in a cubic foot of air). Instruments are also used in air pollution studies, medical research and industrial hygiene.

Gucker [251 - 252] also designed a photometer for rapid measurement of angular patterns from individual particles. A nearly complete 360° scattering pattern is recorded in 20 ms from particles in an aerosol stream flowing through a 0.32 × 0.8 μm laser beam. The instrument has been used to characterize latex spheres in the size range 0.3 to 11 μm [253]. Data are stored on magnetic tapes and the individual curves are fitted to Mie theory diagrams to obtain a 'best fit'.

Optical particle counters based on light scattering by single particles are widely used in aerosol research. In the LASS a fine filament of aerosol is drawn through a laser beam and the scattered light is picked up at a mean angle of 40°. Pulse height is related to particle size by means of a calibration curve size range 0.05 to 0.70 μm at particle concentrations of up to 10$^4$ cm$^{-3}$ [241]. A description of the LASS and a low-angle scattering instrument (LASI) is given by Gebhart et al. [218].

Lasers have replaced the traditional white light source in some recently introduced commercial instruments. The particles are illuminated with a source of radiation nearly 10 times more intense than is possible with the hottest incandescent source known. This results in systems fully capable of sizing particles of 0.1 μm diameter using solid state silicon detectors, 0.08 μm using a photomultiplier and 0.05 μm using a photomult-
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tipler and a curved mirror [187, 205]. Kratel [289] describes the application of an intracavity laser to HEPA filter testing in the size range 0.06 to 3 μm. Allen [267] describes an optical system for asymmetric light-scattering determination by He–Ne laser to determine the size and shape of polystyrene latex, soot and outside aerosol particles. Some of the commercial light-scattering instruments are described below.

![Diagram of the dust counter](image)

**Fig. 3.8 Functional block diagram of the dust counter.**

The Bausch and Lomb dust counter 40-1 uses the near-forward light-scattering principle [189] (figure 3.8). The direct light is captured by a light trap and completely absorbed, and light scattered through an angle of from 24° to 40° is reflected via a parabolic mirror to a photomultiplier. The instrument is notable for its built-in calibration system and is designed to operate in the following size ranges: 0.3, 0.5, 1.0, 2.0, 5.0 and 10 μm in seven increments. Incorporated in the instrument is a dilution system which permits counting at dust concentrations of up to 10⁶ particles ft⁻³. The counting rate is up to 35 particles s⁻¹ at a flow rate of 3 cm³ s⁻¹. Brown [191] added circuitry to the system to allow unattended, automatic measurements to be determined.

The Royco Airborne Dust Counters are available in both modes, forward and right-angle scattering. Model 220, designed for clean room requirements, employs right-angle scatter (figure 3.9). Sampling rate is 47 cm³ s⁻¹ at a maximum concentration of 10⁶ particles m⁻³; counts are recorded of particles larger than 0.5 and 5 μm; other sizes are also available.

Models 225/245 use near-forward scatter. Model 225 is a high-concentration aerosol counter designed to operate at concentrations up to 3.5 x 10⁹ particles m⁻³ at flow rates of 0.01 or 0.10 ft³ min⁻¹ (4.7 or 47 cm³ s⁻¹) and record particles larger than 0.5 μm. Model 245 is a high-flow-rate version designed to operate at concentrations up to 3.5 x 10⁹ particles m⁻³ at a flow rate of 470 cm³ s⁻¹. Model 218 is a portable version operating at concentrations up to 3.5 x 10⁹ particles m⁻³ at a flow rate of 4.7 cm³ s⁻¹.
The calibration of the Royco using cascade impactors was described by Marple and Rubow [73]. Jones and Khaled [192] carried out comparison tests with a thermal precipitator and Jaenicke [193], considered some of its defects. Its uses as a clean air monitor in an operating theatre [194], a filtration monitor [195], for filter testing using diocetyl phthalate [288] and as a size of aerosols have also been described [196].

Climet manufactures a range of aerosol counters, together with two liquid-borne counters, all operating under the same principle. The sampling volume is at the focus of an elliptical mirror (figure 3.10) which accepts the scattered light and focuses it on to a photomultiplier. The light is picked up over the scattering angles 15° to 105° making up a solid angle of 5.1 sr. This maximizes the amount of scattered light picked up and minimizes errors due to particle shape, colour and orientation. Standard particle size ranges are 0.5, 1, 3, 5, 10 μm (0.3 μm available). Particle concentrations of up to 10^9 ft^-3 may be sampled. Sample flow rate is from 1 to 20 ft^3 h^-1. Standard options and accessories are available. The Climet 208 has been successfully used to study the growth of a monodisperse NaCl aerosol as a function of relative humidity [197]. A microcomputer has also been attached to give direct print-out [280].

The Kratel Partoscope model A is a forward white-light-scattering instrument operating at a flow rate of 47 cm^3 s^-1 and a maximum concentration of 300 particles cm^-3. Various size options are available: greater than 0.3 or 0.5 μm and greater than 5 μm; a five-channel model at 0.3, 0.5, 1.5, 3, 5, 10 μm or 0.5, 0.7, 1.4, 3, 5, 10 μm. Model R is a low-flow-rate (5 cm^3 s^-1) version.

The Kratel Aerosol Spectrometer MR is a right-angle scattering collector operating at concentrations up to 10^6 cm^-3 with a lowest detectable size of 0.3 μm. Read-out is digital in two or four channels or multi-channel analyses, minicomputer with printer, plotter or teletype.

The Kratel Partoscopy LA and LC are laser forward-scattering instruments for the sub-micron and respirable range respectively. These are claimed to have advantages over conventional light systems. In particular the use of a 50% beam splitter to produce two image planes for two detectors provides a means of precisely defining a particle's position in the sampling volume. This system also allows complete control of the sensing volume which can be varied by varying the magnification and depth of fluid of the optics.

A low-angle laser light-scattering (LALLS) instrument for particles smaller than 10 μm is also available from Beckman [198].

In the Malvern Instrument's droplet size analyser, Fraunhofer diffraction is applied to droplet size analysis [268]. The light source comprises a 1 mW He-Ne laser and the scattered light is focused on to a multi-element detector. For a given particle the position of the maximum in the diffraction pattern is determined by its size so that the light intensities at different radii from the lens axis are size-dependent. The multi-element detector consists of a series of concentric annuli positioned at the maxima of certain particle diffraction patterns. Their output is monitored to give the size distribution by weight at each of 30 diameters ranging from 5.51 to 472 μm.

Most of the above instruments have not been adopted for routine use in mines because of the stringent power and weight considerations imposed by practical considerations. For these reasons the above principles have been applied by the Centre for Air Environmental Studies in the construction of a battery-driven portable counter [202]. The CAES portable instrument can handle 1500 particles cm^-3 at several size levels with instantaneous readout. Since no light trap is employed a compromise viewing angle of 135° to the incident radiation is used.

The Rotheroe and Mitchell Digital Dust Indicator is a battery-operated, portable monitor of airborne particles and uses the principle of right-angle scattering. The instrument gives the total count above a preset limit (0.3 or 0.5 μm) at an upper concentration level of 100 mg m^-3 or a count rate of 167 s^-1.

Particle Measurement Systems manufacture a range of Knollenberg Aerosol Spectrometer Probes comprising a Classical, Active, Forward and Axially Scattering system. These counters have been examined by Pinnick et al. [281] and their resolution has been found to be significantly less than claimed.

In the HC 15 [290] the aerosol, at velocities in the range 0.2 to 15 m s^-1, passes through a measuring volume where it is illuminated by a halogen lamp. Light scattered at 90° is picked up by a detecting system and the signals from the detecting photomultiplier are passed to a channel analyser where they are measured and stored in 64 channels. The stored sizes are in a logarithmic distribution in the size range 0.3 to 60 μm.

The HIAC automatic portable counter employs light blocking, the attenuation pulses being measured as particles are drawn through the measurement zone (figure 3.11). Five standard models are available with one to five size channels with a claimed size range of from 2 to 9000 μm and flow rates from 20 to 225 000 cm^3 min^-1 depending on the size range.
aerosol, flowing at about 8 cm$^3$ s$^{-1}$, is sheathed in clean air flowing at ten times this rate. The sheath is intended to constrain the particles and prevent contamination of the optics. The aerosol is collected on a membrane filter. Size levels are 0.5, 1.0, 2.0, 5.0, 10.0, > 10 μm at particulate levels between 3.5 × 10$^6$ and 3.5 × 10$^8$ particles m$^{-3}$.

Calibration is by directing light pulses onto the sensor to simulate particles. Prime calibration is carried out at the factory using polystyrene spheres.

Air Technology manufacture a forward-scattering photometer, TDA-2C, for the determination of total atmospheric particulate mass.

Dynac manufacture an airborne particle monitor, M-201C, for measuring airborne particulate contamination in the selectable size ranges > 0.15, 1.0, 3.0, 5.0 μm with an optional > 0.3 μm at flow rates and count levels up to 47 cm$^3$ s$^{-1}$ and 770 particles cm$^{-3}$.

The MRI Integrating Nephelometer has sufficient sensitivity to measure gas scattering; it has also been used to measure aerosol mass concentrations up to 3800 μg m$^{-3}$.

The Digital Dust Indicator is a portable instrument operating with 90° scattered light. Air is drawn through at 10 l min$^{-1}$ and the coarse particles removed by an impaction system. Light pulses from particles passing through the beam are counted by a photomultiplier circuit and recorded.

The Gardner Small Particle Detector depends for its operation on the fact that small particles act as condensation centres for water vapour under suitable conditions. This allows for measurement of particles with radii as small as 10$^{-7}$ cm. Its range is 2 × 10$^2$ cm$^{-3}$ to 10$^7$ cm$^{-3}$.

The GEC condensation nuclei counter and the RAC condensation nuclei counter operate on similar principles.

The Sartorius Spectral Counter is an instrument for quantitative and, under certain conditions, qualitative aerosol analysis. It operates on the principle of scintillation spectrophotometric analysis [250]; the aerosol particles are brought into a heating chamber and on attaining the excitation temperature each particle emits a light impulse which is evaluated by means of an electronic-optical system. The intensity of the impulse is a measure of particle size while the spectral composition shows the chemical composition of the particle. Theoretical and practical considerations show that this instrument is capable of more accurate measurement of aerosol parameters than any other available unit. The unit displays the mass concentration in mg cm$^{-3}$, the particle concentration in particles cm$^{-3}$ and the particle size distribution in ten ranges from 0.01 to 100 μm. Aerosol flow rate is 3 cm$^3$ s$^{-1}$ at a maximum concentration of 20 particles cm$^{-3}$. A selection of monochromators is available for the measurement of single elements only.

In the Sinclair-Phoenix Forward Scattering Photometer, light is brought to a focus at the sample cell, with a diaphragm stop placed in the optical path so that a diverging cone of darkness encompasses the light-collecting lens of the photomultiplier housing. The only light reaching the detector is that scattered in the forward direction by particles in the aerosol under test. The particles are introduced into the light beam through an air inlet and pass through the beam sheathed in clean air. They can then be collected on a molecular filter for subsequent examination. Air flow can be varied up to 1 ft$^3$ min$^{-1}$ (472 cm$^3$ s$^{-1}$). Solids concentration as low as 1 μg m$^{-3}$ may be detected, the mass...
concentration at any instant being displayed on a meter or plotted on a recorder [199]. The Brice-Phoenix Universal Light Scattering Photometer [200, 201] was originally designed to measure scattered light, transmittance, fluorescence and reflectance but it was refined to measure turbidity, angular dependence on Rayleigh ratio, disymmetry and polarization ratio. It operates in the size range 1 to 5000 nm; the light source is a regulated high-pressure mercury vapour lamp in front of which one or more neutral density filters may be stationed. The beam width is adjustable and available wavelengths comprise 365, 405, 436, 546, 578 nm plus 633 nm if the mercury lamp is replaced with a helium-neon source. The instrument has been adapted to determine the particle size distribution of aerosols.

The Sinclair-Phoenix aerosol photometer is a near-forward scattering aerosol photometer employing an axi-symmetric optical arrangement. Light scattered from particles within 4° to 36° of the forward direction indicates concentration. Air is drawn through the instrument at 1 ft² min⁻¹ and is sheathed in clean air. The upper and lower concentration limits are 100 and 10⁴ µg L⁻¹.

The Sartorius aerosol photometer S is a 45° scattering photometer for mass concentration measurement, designed for continuous measurement in the fields of emission control and clean room supervision. The source is a 200 W mercury arc lamp and the direct beam to the photomultiplier is interrupted by a chopper to correct for source intensity variations. Operation is over six orders of magnitude at concentrations from 10⁸ to 5 x 10⁶ particles cm⁻³.

The Leitz Tyndalloscope is a small light-scattering photometer in which the angle of observation is 30°. A chamber is opened to admit ambient air and its intensity matched using crossed polarizing filters and a split eyepiece.

3.8.1 Discussion

The polar light-scattering diagrams for small particles are extremely complex and highly dependent upon relative refractive indices and the wavelength of the incident radiation. The wide adoption of these instruments in the field of health and safety is due to the size dependency being greater in the respirable range. It must be remembered, when using these instruments, that there is no universal light-scattering curve. These instruments have to be calibrated and if they are calibrated with PVC spheres they should be able to size PVC spheres accurately. They will not accurately size spheres having optical properties different from those of PVC. They will not accurately size non-spherical coal dust particles but this raises the thorny problem as to how one defines size for an irregularly shaped particle.

In order to obtain meaningful results one should calibrate using the particles of interest as West does with the HIAC (see section 2.1) and Marple and Rubow [73] with the Royco and Bausch & Lomb.

The errors with light-scattering particle counters have been discussed by Kratel [203] and instruments have been compared by Rimbek and Keaf [204] who find little agreement between them. Whitby and Vomel [205] compared the Royco PC200, the Bausch & Lomb, and the SRI with microscopy and also found significant variations and a tendency to undercount. Heintzenberg [255] compared the two-channel Royco, a condensation counter and a four-channel integrating nephelometer for sizing aerosols in the size range 0.05 to 2 µm.

3.9 Miscellaneous techniques

In the hot-wire anemometer [219], aerosol particles are drawn past a fine, short, hot filament, from which heat is extracted due to impingement on the filament and as a result of turbulence due to the presence of particles. KLD Associates manufacture a version for use in pesticide disbursement, fog and cloud analysis and aerosol monitoring in the size range 1 - 600 µm at counts up to 500 s⁻¹[cited in 254].

A diffusion battery [220, 221] has also been described in which small particles in a gas are subjected to molecular bombardment, which causes them to move in an erratic manner. Airborne particles passing through a narrow capillary tend, therefore, to collide with the capillary walls, and this property may be used for size determination of sub-micrometre particles. A discussion of data interpretation has been given [222] and a comparison with electron microscopy gave reasonable agreement. Knutson [cited in 13] coupled a diffusion battery to a data-processing system which allowed unattended runs of several days with hourly sampling. Kasper et al. [282] describe a six-stage miniature diffusion battery with 'collimated hole structure' plates. The penetration of salt crystals in the size range 0.2 to 0.6 µm was in good agreement with theory. Soderholm [283] gives a brief review of the use of diffusion batteries. Accurate formulae are included for the penetration function of circular tubes and parallel plate cells. A method of obtaining a computer solution is outlined and illustrated with experimental data.

The amplitude of vibration of airborne particles in an intense sound field has been used for determining particle size [223 - 225]. The relationship between the amplitude of particle vibration and particle size has been given by Brandt et al. [223] and suitable equipment has been designed by Schultz and Gucker [224, 225].

A β-absorption impactor aerosol mass monitor has also been described [226]. New techniques include the use of ionization mass [227], gas chromatography [228] and a hot-wire anemometer [229] for aerosol droplets.

The Eberline Model AIM - 3 Air Monitor uses interchangeable crystals to allow either α or β-γ monitoring.

The Radioactive air monitor monitors air with a dual phosphor probe for α and β radiation simultaneously.

A constricted glow discharge has been used for sizing particles in the 10 µm range. The particles are drawn through a 153-µm aperture situated between two electrodes. The magnitude of the perturbation in the discharge current was found to be proportion al to particle size [230].

The time-of-flight aerosol spectrometer shows promise as a method for continuous particle size determination from a few Ångströms up to a micron in diameter [231]. The aerosol is expanded through a nozzle into a vacuum and the particle velocity in the resulting flow reflects the distribution and allows size fractionation.
Mallove and Hinds describe an aerosol disc centrifuge [232]. The time variations of scattered light intensity at fixed radial locations characterize the distribution. They describe the sedimentation–diffusion theory on which the instrument is based and the applied data inversion techniques. Experimental results are presented for a mono- and a polydisperse aerosol. The technique is very rapid ( < 5 min) and ideally suited for liquid droplets.

Some authors [233 - 236] have used holography to measure particle size and velocity. Also, a three-dimensional scanner, for quantitative particle analysis from a real holographic image is commercially available (Optronics Holoscan). The Holographic particle analyser uses a switched ruby rod to produce a hologram of the viewing volume containing the aerosol particles. Particle size, using the hologram, can be studied manually or automatically using television screens. The lower size limit is 0.1 μm.

Specialized sampling devices are also available such as the plutonium dust sampler [137] and the airborne bacteria sampler for sampling bacteria for subsequent counting after incubation (Casella).

Zhuhalov et al. [284] propose a method for the determination of the concentration and the degree of dispersion of sub-micron aerosols using a He-Ne resonator for uniform illumination. The method is applicable to particle radii less than 0.03 μm and concentrations less than 5 × 10^4 cm^-2.

Galli and Madelaine [285] present two size distribution analysers, one based on classical detection principles and the other a photon-counting device. The respective size ranges (μm) and concentrations are: r > 0.2, c > 0.3 × 10^5; 0.03 < r < 0.2, c < 2 × 10^6.

References
5 Breuer, H., et al., ibid., 187.
7 Shah, M.A. et al. (1977), Powder Technol., 18, 53-64.
Sampling and sizing from the atmosphere

4 Particle size, shape and distribution

4.1 Particle size

The size of a spherical homogeneous particle is uniquely defined by its diameter. For a cube the length along one edge is characteristic, and for other regular shapes there are equally appropriate dimensions. With some regular particles, it may be necessary to specify more than one dimension. For example: cone, diameter and height; cuboid, length, width and height.

Derived diameters are determined by measuring a size-dependent property of the particle and relating it to a linear dimension. The most widely used of these are the equivalent spherical diameter. Thus, a unit cube has the same volume as a sphere of diameter 1.24 units, hence this is the derived volume diameter.

If an irregularly shaped particle is allowed to settle in a liquid, its terminal velocity may be compared with the terminal velocity of a sphere of the same density settling under similar conditions. The size of the particle is then equated to the diameter of the sphere. In the laminar flow region, the particle moves with random orientation, but outside this region it orients itself to give maximum resistance to motion so that the free-falling diameter for an irregular particle is greater in the intermediate region that in the laminar flow region. The free-falling diameter, in the laminar flow region, becomes the Stokes diameter. Stokes' equation can be used for spherical particles, up to a Reynolds number of 0.2, at which value it will give a diameter under-estimation of about 2%. Above 0.2 corrections have to be applied. Corrections may also be applied for non-spherical particles, so that the derived diameter is independent of settling conditions becoming purely a function of particle size. These diameters are particularly useful for characterizing suspended particles in the atmosphere and other cases where the settling behaviour of suspended solids is being examined.

For irregular particles, the assigned size usually depends upon the method of measurement, hence the particle sizing technique should, wherever possible, duplicate the process one wishes to control. Thus, for paint pigments, the projected area is important, while for chemical reactants, the total surface area should be determined. The projected area diameter may be determined by microscopy for each individual particle, but surface area is usually determined for a known weight or volume of powder. The magnitude of this surface area will depend on the method of measurement, permeametry, for example, giving a much lower area than gas adsorption. The former gives the surface
area accessible to the gas molecules and, therefore, depends on the size of the gas molecules if the solid contains very small pores.

**Table 4.1 Definitions of particle size**

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Name</th>
<th>Definition</th>
<th>Formula</th>
</tr>
</thead>
<tbody>
<tr>
<td>$d_o$</td>
<td>Volume diameter</td>
<td>Diameter of a sphere having the same volume as the particle</td>
<td>$V = \frac{\pi}{6} d_o^3$</td>
</tr>
<tr>
<td>$d_s$</td>
<td>Surface diameter</td>
<td>Diameter of a sphere having the same surface as the particle</td>
<td>$S = \pi d_s^2$</td>
</tr>
<tr>
<td>$d_{sv}$</td>
<td>Surface volume</td>
<td>Diameter of a sphere having the same external surface to volume ratio as a sphere</td>
<td>$d_{sv} = \frac{d_s^3}{d_o^2}$</td>
</tr>
<tr>
<td>$d_d$</td>
<td>Drag diameter</td>
<td>Diameter of a sphere having the same resistance to motion as the particle in a fluid of the same viscosity and at the same velocity ($d_d$ approximates to $d_s$ when Re is small)</td>
<td>$F_D = \frac{1}{\eta} \frac{d_d^2}{2} \rho f(d_d)$ where $C_D A = f(d_d)$ in $F_D = \frac{3}{2} \frac{d_d g \rho \eta}{Re &lt; 0.2}$</td>
</tr>
<tr>
<td>$d_f$</td>
<td>Free-falling diameter</td>
<td>Diameter of a sphere having the same density and the same free-falling speed as the particle in a fluid of the same density and viscosity</td>
<td>$d_f$</td>
</tr>
<tr>
<td>$d_{Stk}$</td>
<td>Stokes' diameter</td>
<td>The free-falling diameter of a particle in the laminar flow region ($Re &lt; 0.2$)</td>
<td>$d_{Stk} = \frac{d_d^2}{d_d}$</td>
</tr>
<tr>
<td>$d_a$</td>
<td>Projected area</td>
<td>Diameter of a circle having the same area as the projected area of the particle resting in a stable position</td>
<td>$A = \frac{\pi}{2} d_a^2$</td>
</tr>
<tr>
<td>$d_p$</td>
<td>Projected area</td>
<td>Diameter of a circle having the same area as the projected area of the particle in random orientation</td>
<td>Mean value for all possible orientations $d_p = d_o$ for convex particles</td>
</tr>
<tr>
<td>$d_c$</td>
<td>Perimeter diameter</td>
<td>Diameter of a circle having the same perimeter as the projected outline of the particle</td>
<td>$d_c$</td>
</tr>
<tr>
<td>$d_A$</td>
<td>Sieve diameter</td>
<td>The width of the minimum square aperture through which the particle will pass</td>
<td>$d_A$</td>
</tr>
<tr>
<td>$d_F$</td>
<td>Fezzi's diameter</td>
<td>The mean value of the distance between pairs of parallel tangents to the projected outline of the particle</td>
<td>$d_F$</td>
</tr>
<tr>
<td>$d_M$</td>
<td>Martin's diameter</td>
<td>The mean chord length of the projected outline of the particle</td>
<td>$d_M$</td>
</tr>
<tr>
<td>$d_R$</td>
<td>Unrolled diameter</td>
<td>The mean chord length through the centre of gravity of the particle</td>
<td>$E(d_R) = \frac{1}{\pi} \int_0^{2\pi} d_R d\theta_R$</td>
</tr>
</tbody>
</table>

The sieve diameter, for square-mesh sieves, is the length of the side of the minimum square aperture through which the particle will pass, though this definition needs modification for sieves which do not have square apertures. In a sieving operation, such a particle will not necessarily pass through the appropriate mesh, particularly if it will only pass through when presented in a particular orientation as with elongated particles. For all such particles to pass through, the sieving time would approach infinity. There is also a range of aperture sizes in any sieve mesh and certain particles may only pass through the largest apertures.

Microscopy is the only widely used particle-sizing technique in which individual particles are observed and measured. A single particle has an infinite number of linear dimensions, and it is only when they are averaged that a meaningful value results; this is similar for a large number of particles. When a linear dimension is measured parallel to some fixed direction (Martin, Feret, unrolled or shear diameter), the size distribution of these measurements reflects the size distribution of the projected areas of the particles. These are called statistical diameters. Comparing the projected area of the particle with series of circles, gives a diameter which describes that particle for the orientation in which it is measured. In microscopy, this is usually the projected area diameter in stable orientation but, in certain cases, the particle may rest in an unstable position to give a lower value. Some definitions of particle size are given in Table 4.1.

These diameters are usually applied to an assembly of particles and distributions are quoted in terms of the measured or derived diameters. Particles having the same diameter can have vastly different shapes, therefore one parameter should not be considered in isolation.

A single particle can have an infinite number of statistical diameters, hence these diameters are only meaningful when sufficient particles have been measured to give average statistical diameters in each size range.

For a single particle, the expectation of a statistical diameter and its coefficient of variation may be calculated from the following equations [1] (see figure 4.1):

$$E(d_R) = \frac{1}{\pi} \int_0^{2\pi} R d\theta_R$$  \hspace{1cm} (4.1)

$$\sigma_R = \sqrt{\left[E(R^2) - \frac{E^2(R)}{E(R)}\right]}$$  \hspace{1cm} (4.2)

$$E(d_F) = \frac{1}{\pi} \int_0^\pi d_F d\theta_F$$  \hspace{1cm} (4.3)

$$\sigma_F = \sqrt{\left[E(d_F^2) - \frac{E^2(d_F)}{E(d_F)}\right]}$$  \hspace{1cm} (4.3a)
4.2 Particle shape

It is known that particle shape influences such properties as the flowability of powders, packing, interaction with fluids and the covering power of pigments, although little quantitative work has been carried out on these relationships. Qualitative terms may be used to give some indication of the nature of particle shape and some of these, extracted from the British Standard 2955: Glossary of Terms Relating to Powders, are given in Table 4.2.

<table>
<thead>
<tr>
<th>Term</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aicular</td>
<td>needle-shaped</td>
</tr>
<tr>
<td>Angular</td>
<td>sharp-edged or having roughly polyhedral shape</td>
</tr>
<tr>
<td>Crystalline</td>
<td>freely developed in a fluid medium of geometric shape</td>
</tr>
<tr>
<td>Dendritic</td>
<td>having a branched crystalline shape</td>
</tr>
<tr>
<td>Fibrous</td>
<td>regularly or irregularly thread-like</td>
</tr>
<tr>
<td>Flaky</td>
<td>plate-like</td>
</tr>
<tr>
<td>Granular</td>
<td>having approximately an equidimensional irregular shape</td>
</tr>
<tr>
<td>Irregular</td>
<td>lacking any symmetry</td>
</tr>
<tr>
<td>Modular</td>
<td>having rounded, irregular shape</td>
</tr>
<tr>
<td>Spherical</td>
<td>global shape</td>
</tr>
</tbody>
</table>

Such general terms are inadequate for the determination of shape factors that can be incorporated as parameters into equations concerning particle properties where shape is involved as a factor. In order to do this, it is necessary to be able to measure and define shape quantitatively.

In recent years there has been an upsurge of interest in particle shape analysis using pattern recognition techniques [79–81] in which input data are categorized into classes. The potential use of these techniques [82] and the use of the decision function in morphological analysis have been introduced.

There are two points of view regarding the assessment of particle shape. One is that the actual shape is unimportant and all that is required is a number for comparison purposes. The other is that it should be possible to regenerate the original particle shape from the measurement data.

The numerical relations between the various 'sizes' of a particle depend on particle shape, and dimensionless combinations of the sizes are called shape factors. The relations between measured sizes and particle volume or surface are called shape coefficients.

4.2.1 Shape coefficients

There are two especially important properties of particles, surface and volume, and these are proportional to the square and cube respectively of some characteristic dimension. The constants of proportionality depend upon the dimension chosen to characterize the particle; the projected area diameter is used in the following discussion.

\[
S = \pi d_R^2 = \sigma_R d_R^2 = \pi x_1^2
\]  

\[
Volume = \frac{4}{3} \pi d_R^3 = \omega_R d_R^3 = \frac{4}{3} \pi x_1^3
\]
Particle Size Measurement

Volume of particle, \( V = \frac{\pi}{6} a_v^3 = \alpha_{v,a} a_v^3 = x_v^3 \) \hspace{1cm} (4.6)

where \( \alpha_v \) and \( \alpha_a \) are the surface and volume shape coefficients, the additional suffix denoting that the measured diameter is the projected area diameter. The symbol \( x \) denotes size, as opposed to diameter, and includes the shape coefficient. This artifact is found to be very useful for general treatment of data.

The surface area per unit volume (the volume-specific surface) is the ratio \( S \) to \( V \):

\[ S = \frac{\alpha_{v,a}}{A} = \frac{6}{A_{v,v}} = \frac{1}{x_{v,v}} \] \hspace{1cm} (4.7)

Further, the volume-specific surface by microscopy is defined as:

\[ S_{v,a} = \frac{6}{A_a} \] \hspace{1cm} (4.8)

One line of approach to the use of shape coefficients was given by Heywood [2] who recognized that the word 'shape' in common usage refers to two distinct characteristics of a particle. These two characteristics should be defined separately, one by the degree to which the particle approaches a definite form such as a cube, tetrahedron or sphere, and the second by the relative proportions of the particle which distinguish one cuboid, tetrahedron or spheroid from another of the same class.

When three mutually perpendicular dimensions of a particle may be determined, Heywood's ratios [3] may be used:

elongation ratio \( n = L/B \) \hspace{1cm} (4.9)

flakiness ratio \( m = B/T \) \hspace{1cm} (4.10)

where

(a) the thickness \( T \) is the minimum distance between two parallel planes which are tangential to opposite surfaces of the particle, one plane being the plane of maximum stability.

(b) the breadth \( B \) is the minimum distance between two parallel planes which are perpendicular to the planes defining the thickness and are tangential to opposite sides of the particle.

(c) the length \( L \) is the distance between two parallel planes which are perpendicular to the planes defining thickness and breadth and are tangential to opposite sides of the particle.

Consider a particle circumscribed by a rectangular parallelepiped of dimensions \( L \) by \( B \) by \( T \), then:

\[ \text{projected area of the particle} \ A = \frac{\pi}{4} d_v^2 = \alpha_v B L \] \hspace{1cm} (4.11)

where \( \alpha_v \) is the area ratio.

Volume of particle equals projected area by mean thickness:

\[ \alpha_{v,a} a_v^3 = \alpha_v B L p_r T \] \hspace{1cm} (4.12)

where \( p_r \) is the prismoidal ratio (see figure 4.2).

Combining equations (4.11) and (4.12) gives:

\[ \alpha_{v,a} = \frac{\pi\sqrt{\pi}}{8} \frac{p_r}{\sqrt{\alpha_v n}} \] \hspace{1cm} (4.13)

If the particle is equidimensional, i.e., \( B = L = T \) and \( n = m = 1 \), then the volume coefficient takes on a special value \( \alpha_e \) where:

\[ \alpha_e = \frac{\pi\sqrt{\pi}}{8} \frac{p_r}{\sqrt{\alpha_v}} \] \hspace{1cm} (4.14)

Thus, \( \alpha_e \) may be used to define particle form. When the particle is not equidimensional the appropriate value of \( \alpha_{v,a} \) is \( \alpha_e / \sqrt{\alpha_v} \) which substantiates the reasoning given earlier that shape is a combination of the proportions and geometrical form.

Heywood classified particles into tetrahedral, prismoidal, sub-angular and rounded. Values of \( \alpha_{v,a} \) for these classes are given in Table 4.3 [4].

\( \alpha_{v,a} \) can be calculated using equation (4.12) combined with direct observation to determine the shape group into which the particle fits and the values of \( m \) and \( n \). This is practicable down to sizes as small as 5 \( \mu m \) by measurements on the number,
mean size, weight and density of closely graded fractions. Indeed, \( \alpha_{sa} \) may be determined directly by weighing a known number of particles of known mean size.

<table>
<thead>
<tr>
<th>Shape group</th>
<th>( \alpha_{sa} )</th>
<th>( P_r )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Angular</td>
<td>0.5–0.8</td>
<td>0.4–0.53</td>
</tr>
<tr>
<td>Prismatical</td>
<td>0.2–0.9</td>
<td>0.53–0.9</td>
</tr>
<tr>
<td>Sub-angular</td>
<td>0.65–0.85</td>
<td>0.55–0.8</td>
</tr>
<tr>
<td>Rounded</td>
<td>0.72–0.82</td>
<td>0.62–0.75</td>
</tr>
</tbody>
</table>

\( \alpha_{sa} \) is more difficult to determine, but Heywood developed the following relationship on the basis of a large number of experimental measurements:

\[
\alpha_{sa} = 1.57 + C \left( \frac{\alpha_{sa}}{m} \right)^{\frac{4}{3}} \frac{n + 1}{n} \tag{4.15}
\]

in which \( C \) is constant depending upon geometrical form. Table 4.4 shows the values of \( \alpha_{sa} \) and \( C \) for various geometrical forms and also for irregular particles.

<table>
<thead>
<tr>
<th>Shape group</th>
<th>( \alpha_{sa} )</th>
<th>( C )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Geometrical forms</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Tetrahedral</td>
<td>0.328</td>
<td>4.36</td>
</tr>
<tr>
<td>Cubical</td>
<td>0.696</td>
<td>2.55</td>
</tr>
<tr>
<td>Spherical</td>
<td>0.524</td>
<td>1.86</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Approximate forms</th>
<th>( \alpha_{sa} )</th>
<th>( C )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Angular</td>
<td>0.38</td>
<td>3.3</td>
</tr>
<tr>
<td>Prismatical</td>
<td>0.47</td>
<td>3.0</td>
</tr>
<tr>
<td>Sub-angular</td>
<td>0.51</td>
<td>2.6</td>
</tr>
<tr>
<td>Rounded</td>
<td>0.54</td>
<td>2.1</td>
</tr>
</tbody>
</table>

### 4.2.2 Shape factors

If it is required to define the size of a particle by a single dimension, it is usual to do so by expressing the size in terms of one of the diameters defined in Table 4.1. The variation between these diameters increases as the particles diverge more from the spherical shape, and hence shape is an important factor in the correlation of sizing analyses made by various procedures.

One of the earliest defined shape factors is the sphericity \( (\psi_w) \) which was defined by Wadell [5–8] as:

\[
\psi_w = \frac{\text{surface area of a sphere having the same volume as the particle}}{\text{surface area of the particle}} = \left( \frac{d_v}{d_s} \right)^2 \tag{4.16}
\]

At low Reynolds number and with convex particles, the drag diameter equals the surface diameter and the Stokes diameter \( (d_{St}) \) is defined as:

\[
d_{St} = \left( \frac{d_v^2}{d_s} \right)^{1/2}
\]

\[
= \psi_w^{1/4} d_v \tag{4.17}
\]

Further, the surface-volume mean diameter is given by:

\[
d_{sv} = \frac{d_v}{d_s} \frac{d_v^2}{d_v^2} = \psi_w d_v \tag{4.18}
\]

For microscope analysis Laird [19] prefers the definition:

\[
\psi_L = \frac{S_0}{\delta_p} \tag{4.19}
\]

where \( S_0 \) is the surface area of a sphere with a diameter equal to the equivalent diameter and \( \delta_p \) is the surface area of the particle computed from the measured surface area.

For rounded images, whose principal dimensions in two directions at right angles to each other are \( a \) and \( b \), Heywood [2] quotes the semi-empirical formula for the equivalent diameter:

\[
d_e = \left( \frac{4}{\pi} \times 0.77ab \right)^{1/2} \tag{4.20}
\]

For rectangular images the following equation yields a result which is only 1% different from the one calculated above (4.19).

\[
d_e = (ab)^{1/2} \tag{4.21}
\]

Particles rest on microscope slides in the position of greatest stability. Cylindrical particles, of length \( kd \) where \( d \) is the cross-sectional diameter, would be expected to rest with the axis horizontal for \( k > 1 \) and vertical for \( k < 1 \). Laird found that this was so and that a region existed, \( 0.85 > k > 1.5 \), where both orientations were adopted.

For discs or cylinders with \( k < 0.85 \):

\[
\psi_L = \frac{2k}{1 + 2k} \tag{4.22}
\]
For cylinders with \( k > 1.5 \):

\[
\psi_L = \frac{2}{1 + 2k}
\]  

(4.23)

(see Table 4.5). Laird also determined sphericity from sieving and sedimentation studies.

Krumbein's [9, 10] definition of sphericity is:

\[
\psi_w^2 = \left( \frac{C}{B} \right)^{12} \left( \frac{B}{L} \right)^6
\]  

(4.24)

where \( L \) is the longest dimension of the particle, the breadth \( B \) is measured perpendicular to this, and \( C \) is the particle thickness. (Note: these definitions are different to Heywood's.)

<table>
<thead>
<tr>
<th>( k )</th>
<th>( d_v )</th>
<th>( d_s )</th>
<th>( d_{st} )</th>
<th>( \psi_w^{-1} )</th>
<th>( \psi_w^{-1} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.125</td>
<td>0.572</td>
<td>0.791</td>
<td>0.485</td>
<td>1.912</td>
<td>1.620</td>
</tr>
<tr>
<td>0.25</td>
<td>0.721</td>
<td>0.866</td>
<td>0.658</td>
<td>1.443</td>
<td>1.316</td>
</tr>
<tr>
<td>0.50</td>
<td>0.909</td>
<td>1.000</td>
<td>0.867</td>
<td>1.210</td>
<td>1.156</td>
</tr>
<tr>
<td>1</td>
<td>1.145</td>
<td>1.225</td>
<td>1.107</td>
<td>1.145</td>
<td>1.107</td>
</tr>
<tr>
<td>2</td>
<td>1.442</td>
<td>1.581</td>
<td>1.377</td>
<td>1.202</td>
<td>1.148</td>
</tr>
<tr>
<td>4</td>
<td>1.817</td>
<td>2.121</td>
<td>1.682</td>
<td>1.363</td>
<td>1.262</td>
</tr>
<tr>
<td>8</td>
<td>2.289</td>
<td>2.915</td>
<td>2.028</td>
<td>1.622</td>
<td>1.437</td>
</tr>
</tbody>
</table>

Hauser [12] proposed a method of assessing particle shape by comparing the particle with an enveloping rectangle of minimum area. If the rectangle length is \( a \) and its width is \( b \), three characteristics are defined:

The elongation ratio \( \varepsilon = \frac{a}{b} \)  

(4.25)

The bulkiness factor \( y = A/ab \)  

(4.26)

The surface factor \( z = C^2/12.64 \)  

(4.27)

where \( A \) is the projected area of the particle and \( C \) is its perimeter.

Medalia [13] represents the particle in three dimensions as an ellipsoid with radii of gyration equal to those of the particle and defines an anisometry in terms of the ratios of the radii.

Church [14] proposed the use of the ratio of the expected values (see equations 4.3 and 4.4) of Martin's and FeSet's diameters as a shape factor for a population of elliptical particles. Cole [15] introduced an image-analysing computer (the Quantimet 720) to compare longest chord, perimeter and area for large numbers of particles. Many other methods have been proposed and reviewed by Pahl et al. [16], and Davies [17], Beddow [18] and Laird [19].

4.2.3 Applications of shape factors and shape coefficients

If an analysis is carried out by two different techniques, the two results can be brought into coincidence by multiplying by a shape factor provided that particle shape does not change with particle size. For example, if the median size by Coulter analysis is 32 \( \mu m \) and by gravitational sedimentation is 29.2 \( \mu m \), multiplying the sedimentation (Stokes) diameters by 32/29.2 will yield the Coulter distribution.

This ratio is in itself a shape factor, but it can be extended by writing the alternative form of Stokes' diameter (see Table 4.1):

\[
\frac{d_{Stk}}{d_e} = \frac{d_s^3}{d_e} \frac{1}{\psi_w} = \frac{d_v}{d_e} = \left( \frac{29.2}{32} \right)^2
\]

Hence, from equation (4.16):

\[ \psi_w = 0.693 \]

If microscopic examination reveals the form of the particles to be cylindrical, for example, of length \( kd \) where \( d \) is the cross-sectional diameter, then the relationship between \( k \), \( \alpha_{sv} \) and \( \psi \) can be found since:

\[ S = (\frac{1}{4} + k)m^2 = \pi d^2 \]

\[ V = \frac{\pi}{3} k d^3 = \frac{\pi}{2} d^2 \frac{d}{6} \]

Hence, the Coulter diameter, \( d_e = d_v = 3d \sqrt{(\frac{4}{3}k)} \)

the surface diameter, \( d_s = d_v = \sqrt{(k + 0.5)} \)

the Stokes diameter, \( d_{Stk} = \frac{d_v^3}{d_{Stk}} = \left( \frac{9k^2}{2(1 + 2k)} \right)^{1/4} \)

the sphericity, \( \psi_w = \frac{(18k^2)^{1/3}}{(1 + 2k)} \)

the surface-volume shape coefficient from sedimentation data,

\[ \alpha_{sv, Stk} = \left( \frac{2 + 4k}{k} \right) \left( \frac{d_{Stk}}{d} \right) = \left( \frac{3}{k} \right)^{1/2} \left[ 2(1 + 2k) \right]^{3/4} \]

the surface-volume shape coefficient from Coulter data,

\[ \alpha_{sv, c} = \left( \frac{2 + 4k}{k} \right) \left( \frac{d_v}{d} \right) = \left( \frac{3}{2k^2} \right)^{1/3} (2 + 4k) \]
It can be shown that:

\[ \frac{\alpha_{\text{SV}}}{6} = \frac{1}{\psi_{w}} \]

Data, for unit diameter cylinders, are presented in Table 4.5. Data for other shapes are presented in Tables 4.6 and 4.7. From these it can be seen that \( \alpha_{\text{SV}} \) is both at a minimum when the shape is most compact (\( k = 1 \)) and increase as the particles become rod-shaped (\( k > 1 \)) or flaky (\( k < 1 \)).

For the numerical illustration, when \( \psi_{w}^{-1} = 1.44 \), \( \alpha_{\text{SV}}/6 = 1.32 \) and \( k = 0.25 \) or 5.5. A visual examination will reveal whether the particles are flaky or rod-shaped.

### Table 4.6 Calculated values of shape coefficients

<table>
<thead>
<tr>
<th>Form</th>
<th>Proportions</th>
<th>Linear dimension used as ( d_{r} )</th>
<th>( \alpha_{b,r} )</th>
<th>( \alpha_{v,r} )</th>
<th>( \alpha_{\text{SV},r} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sphere</td>
<td>1 : 1 : 2</td>
<td>diameter</td>
<td>3.14</td>
<td>0.52</td>
<td>6.00</td>
</tr>
<tr>
<td>Spheroid</td>
<td>1 : 2 : 2</td>
<td>minor axis</td>
<td>5.37</td>
<td>1.05</td>
<td>5.13</td>
</tr>
<tr>
<td></td>
<td>1 : 1 : 4</td>
<td>minor axis</td>
<td>10.13</td>
<td>2.09</td>
<td>4.43</td>
</tr>
<tr>
<td>Cylindrical</td>
<td>1 : 4 : 4</td>
<td>minor axis</td>
<td>28.50</td>
<td>8.38</td>
<td>3.40</td>
</tr>
<tr>
<td>Ellipsoid</td>
<td>1 : 2 : 4</td>
<td>shortest axis</td>
<td>15.86</td>
<td>4.19</td>
<td>3.79</td>
</tr>
</tbody>
</table>

### Table 4.7 Measured values of surface-volume shape coefficient

<table>
<thead>
<tr>
<th>Material</th>
<th>Approximate sizes</th>
<th>( \alpha_{SV} )</th>
<th>Specific surface method</th>
<th>Particle size method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alumina</td>
<td>15-45</td>
<td>16</td>
<td>permeametry</td>
<td>microscope</td>
</tr>
<tr>
<td>Coal</td>
<td>15-90</td>
<td>12-17</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Dolomite</td>
<td>25-45</td>
<td>11</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Silica</td>
<td>15-70</td>
<td>11</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Tungsten oxide</td>
<td>15-45</td>
<td>14</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Coal</td>
<td>15-90</td>
<td>10-12</td>
<td>permeametry</td>
<td>Coulter counter (( d_{a} ))</td>
</tr>
<tr>
<td>Coal</td>
<td>15-70</td>
<td>9</td>
<td>light weight</td>
<td></td>
</tr>
<tr>
<td>Diamond</td>
<td>0.5-12</td>
<td>8</td>
<td>extinction count (( d_{i} ))</td>
<td></td>
</tr>
<tr>
<td>Quartz</td>
<td>0.5-10</td>
<td>9</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

(Table 4.6 and 4.7 are from BS 4359 (1970): Part 3, reproduced by permission of the British Standards Institution, 2 Park Street, London W.I, from whom copies of the complete standard may be obtained.)

Example

Consider two cuboids of similar shape but different sizes, i.e. side lengths 1 : 2 : 3 and 2 : 4 : 6:

- maximum projected area: \( 6 + 24; A = 30 \)
- total surface area: \( 22 + 88; S = 110 \)
- total volume: \( 6 + 48; V = 54 \)
- projected area diameter: \( d_{a} = \frac{\pi}{4} \frac{a_{2}^{2}}{a_{1}} \)
- mean surface diameter: \( \bar{d}_{a} = \frac{4}{S} \)
- surface shape coefficient: \( S_{a} = \frac{a_{1}}{a_{2}} \)
- mean volume diameter: \( V = \frac{\pi}{6} \frac{a_{3}^{3}}{a_{2}} \)
- volume shape coefficient: \( \bar{d}_{a} = \frac{4}{S} \frac{a_{2}^{2}}{a_{1}} \)
- surface-volume mean diameter: \( d_{sv} = \frac{d_{a}^{3}}{a_{2}} \)
- volume-specific surface: \( S_{a} = \frac{S}{V} = \frac{a_{1}}{d_{sv}} \)
- volume-specific surface by microscopy: \( S_{a} = \frac{6}{d_{a}} \)

(See BS 4359 (1970): Part 3, for further examples.)

The surface-volume shape coefficient has been determined for quartz and silica from surface area measurements using nitrogen adsorption giving \( 14 < \alpha_{SV} < 18 \) with no significant variation with particle size. Fair and Hatch [21] found that by measuring smoothed surfaces, values of \( \alpha_{SV,a} \) as low as 7 were found (\( \alpha_{SV,a} = 6 \) for spheres). Crowl [22] found that with Prussian blue, the specific surface by nitrogen adsorption applied to the primary particles of which each single particle is made up (see figure 4.3). With red iron oxide, however, the specific surface applied to the single primary particle.

The mean volume shape may be determined from a knowledge of the number, mean size, weight and density of the particles composing a fraction graded between close limits. Further, if the surface area is determined by permeametry, a surface shape may be evaluated though this will differ from that obtained from the gas adsorption surface area. Hence, when any shape is quoted, the method of obtaining it should also be given.

Ellison [23] obtained the value 0.9 for the ratio of the sizes of silica particles determined by settling experiments and mounted in agar in random orientation. Hodkinson
Particle size, shape and distribution

[24] found, from measurements of quartz particles by light scattering, a diameter ratio of 0.8 between particles in a liquid suspension and settled particles. Cartwright [57] attempted to find the magnitude of the differences in mean projected diameters between particles of quartz in random and stable orientation by microscopy. He used four different mounting techniques and found no significant differences. This, he attributed to the difficulty in mounting particles in random orientation. These factors for the mean ratio of projected diameter for random and stable orientation are indicative of the properties of the powder and are therefore of use to the analyst.

Respirable coal mine dust samples from three different US mines were classified into four fractions using a Bahco [68]. Shape factors were determined as ratios of the following diameters from microscopy:

\[ d_a = \left( \frac{\Sigma n d_a^2}{\Sigma n} \right)^{\frac{1}{2}} \]

\[ A_p \] from photosedimentation with correction for extinction coefficient,

\[ N_w \] from microscopy:

\[ d_p = \left( \frac{4A_p}{\pi N_w} \right)^{\frac{1}{2}} \]

\[ \left( A_p = \frac{\pi}{4} \sum n d_p^2 \right) \]

\[ S_w \] from krypton gas adsorption, \[ N_w \] from microscopy:

\[ d_{BET} = \left( \frac{S_w}{\pi N_w} \right)^{\frac{1}{2}} \]

\[ p \] from density measurement, \[ N_w \] from microscopy:

\[ d_w = \frac{3}{2pA_p} \]

where \[ N_w \] = number of particles/unit weight

\[ A_p \] = projected area of particles, in random orientation, per unit weight. See Table 4.1 for definitions of other symbols.

It was postulated that a relationship might exist between shape and the incidence of pneumoconiosis.

A method, for shape determination, has been described in which the image analysis system consists of an Optronics scanning microdensitometer and a computer program [58]. It performs quantitative measurements of particle size distribution, shape and edge texture from photographs, where shape is defined as perimeter divided by projected area. A full analysis takes about three and a half man-hours and its most successful application has been in the analysis of silica sands in deep bed air filters for the removal of radioactive particulates. A relationship was found between collection efficiency and edge texture [59].

---

Fig. 4.3 Electron photomicrographs of two paint pigments, showing how particles can be aggregates of finer particles (Crowel [22]). (a) A single particle of Prussian blue about 1 μm in diameter. The nitrogen adsorption surface area is 61.3 m² g⁻¹ from which the surface-volume mean diameter is 0.051 μm. This is seen to be the diameter of the individual primary particles of which the aggregate is made up. Similarly, the micronized Prussian blue (b) has approximately the same surface-volume mean diameter. (c) With the red oxide, the diameter is 0.21 μm which is approximately the same as the solid particle seen in the micrograph.
4.2.4 Shape indices

Tsubaki and Jimbo [1, 25, 26] argue that many of the proposed shape factors had little practical applicability to the analysis of real powders until the advent of electronic techniques and the computer. They defined six shape indices based on ratios of the following diameters (see Table 4.1): \(d_e, d_v, d_F, d_R\). The shape indices are: \(\psi_{se}, \psi_{sf}, \psi_{sr}, \psi_{eF}, \psi_{eR}, \psi_{FR}\), where, for example, \(\psi_{se} = d_v/d_e\). They defined the statistical diameters and coefficients of variation according to equations (4.1) to (4.4).

Furthermore, the elongation was used for study.

\[
Z = \frac{d_{FR}}{d_{Fmin}}
\]

(4.28)

where \(d_{Fmin}\) is the minimum value of the Feret diameter and \(d_{F_{3/2}}\) is the diameter perpendicular to this.

They later added three more indices \(\psi_{F3}, \psi_{Stk}\) and \(\kappa\). The arithmetic average of breadth and length is defined as follows:

\[
d_F = \frac{1}{2} (d_{Fmin} + d_{F_{3/2}})
\]

(4.29)

The dynamic shape factor \(\kappa\) is defined as the ratio of the resistance to motion of a given particle divided by the resistance of a spherical particle of the same volume. When the particle is settling under laminar flow conditions:

\[
\kappa = \left(\frac{d_v}{d_{Stk}}\right)^2
\]

(4.30)

Since this is a squared term which for comparison purposes they wished to reduce to unit power they introduced the shape factor \(\psi_{Stk} = d_{Stk}/d_v\).

[Note \(\kappa = \psi_{Stk}^{1/2}\), where \(\psi_{Stk}\) is Wadell’s sphericity factor.]

For non-re-entrant particles, according to Cauchy’s theorem, \(d_e = E(d_F)\) and \(\psi_{eF}\) has a maximum value of 1.0 for circles, rectangles and other convex shapes; it is therefore very useful for indicating the extent of concavities.

\(\psi_{RE}, \psi_{RF}, \psi_{ER}, \psi_{FR}, \psi_{eF}, \psi_{eR}\) and \(Z\) were found to mainly show the slimness of the particles, the best indicators being \(\psi_{eF}\) and \(Z\) in that order.

\(\psi_{He}\) and \(\psi_{Re}\) were found to correspond poorly with particle morphology.

4.2.5 Shape regeneration by Fourier analysis

Briefly, this method consists of finding the particle outline and its centre of gravity from which a polar co-ordinate system is set up. Standard Fourier transform techniques are then used to generate the Fourier coefficients \(A_n\) and their associated angles \(\theta_n\).

In 1969 Schwarcz and Shane [27] published a paper where Fourier transforms were used to analyze beach sand silhouettes. In 1969 and 1970 Moley [28, 29] presented papers in which fast Fourier transforms were used to process particle silhouette as a signal and this work was extended in 1977 [30, 31]. One of their main conclusions is that particles have ‘signatures’ which depend on \(A_n\) and not on \(\theta_n\) and they propose the equation:

\[
A_n = A_1 \left(\frac{1}{n}\right)^s
\]

(4.31)

A plot of \(\log A_n\) against \(\log n\) yields a straight line which has a slope \(s\) which depends on particle shape. Rounder particles having lower slopes. Beddow [18] showed how a number of particle silhouette shapes could be analysed and reproduced by Fourier transforms. Gotch and Finney [32] proposed a mathematical method for expressing a single, three-dimensional body by sectioning as an equivalent ellipsoid having the same volume, surface area and average projected area as the original body.

4.2.6 Fractal dimension characterization of textured surfaces

Mandelbrot has introduced the term ‘fractal dimension’ to describe curves which have no unique perimeter. In his book [33] he describes how Richardson estimated the length of the coast line of various countries by stepping along a map of the coast line with a pair of dividers. He found that the estimated perimeter \((P_k)\) tends to increase without limit as the step size \(\lambda\) decreases. He concluded that a polygon of side \(n\) \((n = \text{number of steps})\) approximating a coast line would have \(\lambda^{1-D}\) sides. The perimeter estimate would be:

\[
P_k = n\lambda
\]

(4.32)

\[
P_k = k\lambda^{1-D}
\]

(4.33)

Hence a plot of \(\log P_k\) against \(\log \lambda\) will have a slope \(1 - D\). The parameter \(D\) was found to be characteristic of the particular coast line measured and was called by Mandelbrot the fractal.

Kaye [34] demonstrated that this technique could be applied to the determination of the ruggedness of carbon black agglomerates. Another procedure he utilized was to study the frequency of intersections made by the boundary of the profile with a grid placed randomly under the profile using grids with a range of grid sizes. A third procedure he adopted was to determine the number of squares on the particle profile, accepting those that were largely inside the profile and rejecting others. The efficiencies of the three techniques were found to be in the order that they are presented above.
His main conclusion was that this technique was too time-consuming for manual operation and he recommended the use of image-animating computers, a recommendation that was taken up by Flook [35] using a Quantimet 720. Flook considered the perimeter to be made up of a series of closely spaced points. A series of circles of radius \( \lambda \) is drawn up with their centres on each of the points in turn thus describing a path of width \( 2\lambda \) covering the curve. The area of the path divided by its width gives an estimate of its length. The process is repeated with decreasing \( \lambda \).

This method was applied to a circle which, being a Euclidean curve, has a fractal equal to unity. Two mathematical models examined consisted of geometrically constructed islands taken from Mandelbrot's book. The first was a Triadich Koch Island and the second a Quadric Koch Island (figure 4.4). Measurements were also made on a typical carbon black aggregate and a simulated carbon floc, taken from a paper by Medalia [36]. Excellent agreement was found between experimental and theoretical results.

4.3 Determination of specific surface from size distribution data

4.3.1 Number distribution

Let particles of size \( d_{x,r} \) constitute a fraction \( m_r \) of the total number \( N \) so that:

\[
\begin{align*}
  m_r &= \frac{n_r}{N} \quad \text{and} \quad N = \Sigma n_r \\
  S &= \alpha_{x,x} N \Sigma m_r d_{x,r}^2 \\
  V &= \alpha_{v,x} N \Sigma m_r d_{x,r}^3 \\
  S_r &= \alpha_{sv,x} \frac{\Sigma m_r d_{x,r}^2}{\Sigma m_r d_{x,r}^2} 
\end{align*}
\]

(4.34)

4.3.2 Surface distribution

Let particles of size \( d_{x,r} \) constitute a fraction \( t_r \) of the total surface \( S \) so that:
\[ t_r = \frac{S_r}{S} \quad \text{and} \quad S = \sum S_r \]

\[ S_t = \alpha_{\text{eq}} \quad \frac{N_r \rho_{\text{sp}}^2}{t_r} \]

\[ V = \frac{S}{\alpha_{\text{eq}} \cdot x} \quad \theta t_r d_{x,r} \]

\[ S_v = \frac{\alpha_{\text{eq},x}}{\sum \theta t_r d_{x,r}} \quad (4.35) \]

4.3.3 Volume distribution

Let particles of size \( d_{x,r} \) constitute a fraction \( q_r \) of the total volume \( V \) so that:

\[ q_r = \frac{V_r}{V} \quad \text{and} \quad V = \sum V_r \]

\[ V q_r = \alpha_{\text{eq}} \quad N r \rho_{\text{sp}}^2 \]

\[ S_v = \alpha_{\text{eq}} \quad \sum \frac{q_r}{d_{x,r}} \quad (4.36) \]

Equation (4.34) is used whenever a number count is taken and yields a specific surface if the shape coefficient is known and vice-versa. If \( \alpha_{\text{eq}} \) is assumed equal to 6 (this is assuming spherical particles) a specific surface is obtained. For microscope counting by projected area this is written \( S_v, x \).

Similar arguments apply to equations (4.35) and (4.36). The latter is frequently applied to sieve analyses where \( q_r \) is the fractional weight residing between two sieves of average aperture \( d_{A,r} \).

4.4 Particle size distribution transformation between number, surface and mass

There are several methods of particle size measurement in which the raw data are collected in the form of a number distribution: microscopy, electrical and light-sensing zone methods are three widely used examples.

Transition is simple if one obtains a straight line on log-probability paper since the graphs for surface and weight are straight lines, parallel to the number distribution, but with the 50% value, corresponding to the surface mean \( x_{ss} \) and volume mean \( x_{sv} \), displaced (equations 4.129 and 4.135).

Considerable difficulties are presented if the log-normal law does not hold. The obvious way of calculating the proportion of surface or volume corresponding to particular particle diameters consists of weighing by the corresponding squared or cubed diameters [76]. The results are then summed to yield the cumulative surface or weight distribution. Conversion from number to surface or volume to volume is best carried out graphically, in order to smooth out experimental errors, as described in the section on photo-sedimentation. Nomograms have been developed to reduce the tedious and possible calculation errors involved in number to weight conversion [72, 73]. However, it is not in general likely that the conversion from number to weight will be accurate on the following grounds:

(1) The weighted proportions are too sensitive to sample fluctuations since the method requires the raising of experimental errors to higher powers. (In a 1 to 10 \( \mu \)m distribution an error of one 10 \( \mu \)m particle has the same weighting as an error of a thousand 1 \( \mu \)m particles.)

(2) The grouping of the observed diameters into frequency intervals, which in practice may be broad and non-uniform, makes an accurate estimation of the average for each size range impossible.

The mean diameter for each range is usually taken as the arithmetic mean \( \bar{x} = \frac{x_1 + x_2}{2} \) or less frequently the geometric mean \( \bar{x} = \sqrt{x_1 \cdot x_2} \).

Thus, the fraction by weight of particles larger than \( D_r \) is given by:

\[ W_r = \sum \frac{D_{\max}}{D_r} D^3 dN \]

where \( \sum D^3 dN = 1 \), i.e. the distribution is normalized.

Integration by parts, however, yields the following equivalent relationship [74, 75]:

\[ W_r = D_{\max}^3 N_r + 3 \frac{D_{\max}^2}{D_r} D^2 dN dD \]

\[ = D_{\max}^2 N_r + 3 \left( (D_{\max}^2 + D_{\max}^2 + 1) \left( \frac{D_{\max}^2 + 1 - D_r}{2} \right) + (D_{\max}^2 + 1 + D_{\max}^2 + 2) \left( \frac{D_{\max}^2 + 2 - D_r}{2} \right) \right) + \ldots \]  

(4.38)

This is applied in Table 4.8 and the results are compared with weighting with cubed diameters.

In using this method the particle counts may be classified into fairly broad intervals thereby reducing experimental time. A computer program for carrying out this conversion has also been published [77]. The conversion is much more accurate, however, if the procedure laid down in the chapter on microscopy is followed. The errors are also reduced with electronic counting devices where the total count is large and the size intervals are small.
Particle Size Measurement

Conversion from weight to surface (e.g., sieving) may be carried out using the following expression:

\[ \Delta S = \sum_{r=1}^{n+1} \frac{\Delta W_r}{d_{Ar}} \]  

(4.39)

where \( \Delta S \) is the relative surface of a weight of powder \( W_r \) residing between sieve sizes \( d_{Ar+1} \) and \( d_{Ar-1} \).

<table>
<thead>
<tr>
<th>Particle size (( D ))</th>
<th>Cumulative number frequency</th>
<th>Cumulative weight frequency</th>
<th>Cumulative percentage oversize</th>
<th>( \Delta N \cdot D^{-2} )</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>( 50 = D_m )</td>
<td>( 0 = N_m )</td>
<td>( 0 = W_m )</td>
<td></td>
<td>.0</td>
<td></td>
</tr>
<tr>
<td>( 45 = D_{m-1} )</td>
<td>( 1 = N_{m-1} )</td>
<td>( 850.5 = W_{m-1} )</td>
<td>6.6</td>
<td>.6860</td>
<td>6.4</td>
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<tr>
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<td>.4913</td>
<td>11.0</td>
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<td>1876.5</td>
<td>14.6</td>
<td>.3375</td>
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</tr>
<tr>
<td>30 = D_r</td>
<td>7 = N_r</td>
<td>3021</td>
<td>23.6</td>
<td>.8788</td>
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<td>12810</td>
<td>100.0</td>
<td>41</td>
<td>100.0</td>
</tr>
</tbody>
</table>

\( \phi \), the frequency function = \( \sum dN \) for a number distribution

\( = \sum x dN \) for a size distribution

\( = \sum x^2 dN \) for an area distribution

\( = \sum x^3 dN \) for a volume or weight distribution where \( dN \)

is the percentage of the total number of particles lying in the size range \( x_1 \) to \( x_2 \).

The most commonly occurring value, the mode, passes through the peak of the relative frequency curve, i.e., it is the value at which the frequency density is a maximum. The median line divides the area under the curve into equal parts, i.e., it is the 50% size on the cumulative frequency curve. The vertical line at the mean passes through the centre of gravity of a sheet of uniform thickness and density cut to the shape of the distribution. Hence, for the mean, the moment of the sum of all the elementary areas of thickness \( \delta x \) about the ordinate equals the sum of all the moments:

\[ \bar{x} \sum \frac{d\phi}{dx} \delta x = \sum x \frac{d\phi}{dx} \delta x \]  

\[ \bar{x} = \frac{\sum x d\phi}{\Sigma d\phi} \]  

(4.40)

For a weight distribution \( d\phi = x^3 dN \) giving:

\[ \bar{x} = \frac{\sum x^4 dN}{\Sigma x^3 dN} \]  

(4.41)

4.5 Average diameters

The purpose of an average is to represent a group of individual values in a simple and concise manner in order to obtain an understanding of the group. It is important, therefore, that the average should be representative of the group. All average diameters
Particle size, shape and distribution

The mode and the median may be determined graphically but the above summation has to be carried out for the determination of the mean. However, for a slightly skewed distribution, the approximate relationship mean — mode = 3 (mean — median) holds. For a symmetrical distribution, they all coincide. In the illustration, the values are: mode = 15.0; median = 17.2; yielding mean = 18.2, as compared with the summed value of 18.47 (see Table 4.10).

Table 4.10 Relative percentage frequency distribution: tabular calculation of mean size

<table>
<thead>
<tr>
<th>Particle size range</th>
<th>Interval size</th>
<th>Average size</th>
<th>Percentage in range</th>
<th>Percentage per micrometre</th>
</tr>
</thead>
<tbody>
<tr>
<td>( x_1 ) to ( x_2 )</td>
<td>( dx )</td>
<td>( x )</td>
<td>( dp )</td>
<td>( dp/dx )</td>
</tr>
<tr>
<td>0 to 5</td>
<td>5</td>
<td>2.5</td>
<td>1.4</td>
<td>0.3</td>
</tr>
<tr>
<td>5 to 9</td>
<td>4</td>
<td>7.0</td>
<td>8.0</td>
<td>2.0</td>
</tr>
<tr>
<td>9 to 11</td>
<td>2</td>
<td>10.0</td>
<td>8.6</td>
<td>4.3</td>
</tr>
<tr>
<td>11 to 14</td>
<td>3</td>
<td>12.5</td>
<td>14.0</td>
<td>4.7</td>
</tr>
<tr>
<td>14 to 17</td>
<td>3</td>
<td>15.5</td>
<td>17.5</td>
<td>5.8</td>
</tr>
<tr>
<td>17 to 20</td>
<td>3</td>
<td>18.5</td>
<td>14.5</td>
<td>4.8</td>
</tr>
<tr>
<td>20 to 23</td>
<td>3</td>
<td>21.5</td>
<td>12.0</td>
<td>4.0</td>
</tr>
<tr>
<td>23 to 28</td>
<td>5</td>
<td>25.5</td>
<td>12.0</td>
<td>2.4</td>
</tr>
<tr>
<td>28 to 33</td>
<td>5</td>
<td>30.5</td>
<td>6.0</td>
<td>1.2</td>
</tr>
<tr>
<td>33 to 41</td>
<td>8</td>
<td>37.0</td>
<td>4.0</td>
<td>0.5</td>
</tr>
<tr>
<td>41 to 50</td>
<td>9</td>
<td>45.5</td>
<td>1.4</td>
<td>0.2</td>
</tr>
<tr>
<td>50 to 60</td>
<td>10</td>
<td>55.0</td>
<td>0.5</td>
<td>0.1</td>
</tr>
</tbody>
</table>

\[ \sum dp \] 1847

Mean size = \[ \frac{\sum x dp}{\sum dp} \] = 18.47

The characteristics of a particle distribution are its total number, length, area and volume. A system of unequally sized particles may be represented by a system of uniformly sized particles having two, and only two, characteristics of the original distribution. The size of the particles in the uniform system is then the mean size of the non-uniform system with respect to these two properties.

The sizes may be expressed mathematically by dividing the system of particles into small intervals of size \( \delta x \) with assumed diameters of \( x_1, x_2, \ldots \). The symbol \( x \) is used because the method of measurement for individual particles is not specified, and all particles are assumed to have the same shape. Let the numbers of particles in these groupings be \( \delta N_1, \delta N_2, \ldots \) respectively. Then the aggregate length, surface and volume of the particles in each grouping are \( x \delta N, x^2 \delta N \) and \( x^3 \delta N \) and the total for the system is the summation of these expressions. Table 4.11 is a summary of the mathematical expressions for the various mean diameters [3].

The method of sizing may also be incorporated into the symbol. Hence, for particle sizing by microscopy, the arithmetic mean diameter becomes \( \bar{d}_a, N_{LV} \). The surface-volume diameter calculated from the results of a sedimentation experiment is \( \bar{d}_{SVK, SV} \).
The mean value of a cumulative weight percentage curve obtained by sieving would be $d_A$, $v_m$ or $d_A$, $w_m$.

Table 4.11 Definitions of mean diameters

| Number, length mean diameter | $x_{NL} = \frac{\sum dL}{\sum dN} = \frac{\sum x dN}{\sum dN}$ |
| Number, surface mean diameter | $x_{NS} = \sqrt{\frac{\sum dS}{\sum dN}} = \sqrt{\frac{\sum x^2 dN}{\sum dN}}$ |
| Number, volume mean diameter | $x_{NV} = \sqrt[3]{\frac{\sum dV}{\sum dN}} = \sqrt[3]{\frac{\sum x^3 dN}{\sum dN}}$ |
| Length, surface mean diameter | $x_{LS} = \frac{\sum dS}{\sum dL} = \frac{\sum x^2 dN}{\sum x dN}$ |
| Length, volume mean diameter | $x_{LV} = \sqrt[3]{\frac{\sum dV}{\sum dL}} = \sqrt[3]{\frac{\sum x^3 dN}{\sum x dN}}$ |
| Surface, volume mean diameter | $x_{SV} = \frac{\sum dV}{\sum dS} = \frac{\sum x^2 dN}{\sum x^2 dN}$ |
| Volume, moment mean diameter | $x_{VM} = \frac{\sum dM}{\sum dV} = \frac{\sum x dW}{\sum x^2 dN}$ |
| Weight, moment mean diameter | $x_{WM} = \frac{\sum dM}{\sum dW} = \frac{\sum x dN}{\sum x^2 dN}$ |

Consider the system illustrated (figure 4.7) consisting of one particle of size 1, 2, 3, 4, 5, 6, 7, 8, 9, 10. Hence ten particles, each of length 5.50, will have the same total length as the original distribution (Table 4.12). Similarly ten particles, each of length 6.21, will have the same total surface as the original distribution.

![Fig. 4.7 The homogeneous distribution that represents in number and length a heterogeneous distribution of ten particles of size 1 to 10 with unit separation in size.](image)

Each of these mean diameters characterizes the original distribution in two properties only. For example, the length-surface mean diameter is 7.00. Therefore, the uniform system contains $N = (L/x) = (S/x^2)$, which is 7.87 in each case. Hence, the uniform system consists of 7.87 particles, each of length 7.00. Thus, the total length and the total surface of the particles are the same as in the original distribution, but the total number, volume and moment are all different, e.g. $V = x^2 N = 343 \times 7.87 = 2700$.

Table 4.12

<table>
<thead>
<tr>
<th></th>
<th>$x_{NL}$</th>
<th>$x_{LS}$</th>
<th>$x_{VM}$</th>
<th>$x_{SV}$</th>
<th>$x_{LV}$</th>
<th>$x_{MV}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$x_{NL}$</td>
<td>5.50</td>
<td>7.00</td>
<td>3025</td>
<td>55</td>
<td>25335</td>
<td></td>
</tr>
<tr>
<td>$x_{LS}$</td>
<td>6.21</td>
<td>7.43</td>
<td>385</td>
<td>55</td>
<td>25335</td>
<td></td>
</tr>
</tbody>
</table>

The arithmetic mean is the sum of the diameters of the separate particles divided by the number of particles, it is most significant when the distribution is normal:

$$x_A = x_{NL} = \frac{\sum x dN}{\sum dN}$$

The geometric mean is the $n$th root of the product of the diameters of the $n$th particle examined; it is of particular value with log-normal distributions:

$$x_g = (\prod x dN)^{1/N}$$

$$N \log x_g = \Sigma d N \log x$$

$$\log x_g = \frac{\Sigma d N \log x}{N}$$

The harmonic mean is the number of particles divided by the sum of the reciprocals of the diameters of the individual particles; this is related to specific surface and is of importance where surface area of the sample is concerned [37]:

$$x_H = \frac{\sum d N}{\sum d N/x}$$

4.6 Particle dispersion

The spread of the distribution data may be expressed in terms of the range, i.e. the difference between the minimum and maximum sizes; the interquartile range ($25^{th}$ to $75^{th}$), i.e. the difference between the 75% and 25% sizes; an interpercentile range, e.g. ($20^{th}$ to $80^{th}$), the difference between the 80% and 20% sizes or the standard deviation.

The least significant of these is the first, since a stray oversize or undersize particle can greatly affect its value. The most significant is the last, since every particle has a weighting which depends on the difference between its size and the mean size.
Particle Size Measurement

The standard deviation \( \sigma \) is defined as:

\[
\sigma = \sqrt{\frac{\sum (x - \bar{x})^2 \Delta \phi}{\sum \Delta \phi}} \quad (4.46)
\]

Hence:

\[
\sigma^2 = \frac{\sum x^2 \Delta \phi}{\phi} - \bar{x}^2 \quad (4.47)
\]

where \( \sigma^2 \) is called the variation.

4.7 Methods of presenting size analysis data

An example of the tabular method of presenting size distribution data is shown in Table 4.13. The significance of the distribution is more easily grasped when the data are presented pictorially, the simplest form of which is the histogram. The data in Table 4.13 give the size grading of 1000 particles in 12 class intervals which are in a geometric progression. The choice of class widths is of fundamental importance, the basic requirement being that the resolution defined as the class interval divided by the mean class size should be kept fairly constant. With narrowly classified powders, an arithmetic distribution is acceptable but it is more normal to use a geometric progression.

Table 4.13 Size data

<table>
<thead>
<tr>
<th>Particle size range</th>
<th>Interval size</th>
<th>Average size</th>
<th>Number frequency in range ( dN )</th>
<th>Percentage in range ( \frac{d\phi}{dx} )</th>
<th>Percentage per micron ( \frac{d\phi}{d \log x} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>( x ) to ( x_{1} )</td>
<td>( dx )</td>
<td>( x )</td>
<td>( dN )</td>
<td>( \frac{d\phi}{dx} )</td>
<td>( \frac{d\phi}{d \log x} )</td>
</tr>
<tr>
<td>1.4 to 2.0</td>
<td>0.6</td>
<td>1.7</td>
<td>1</td>
<td>0.1</td>
<td>0.2</td>
</tr>
<tr>
<td>2.0 to 2.8</td>
<td>0.8</td>
<td>2.4</td>
<td>4</td>
<td>0.4</td>
<td>0.5</td>
</tr>
<tr>
<td>2.8 to 4.0</td>
<td>1.2</td>
<td>3.4</td>
<td>22</td>
<td>2.2</td>
<td>1.8</td>
</tr>
<tr>
<td>4.0 to 5.6</td>
<td>1.6</td>
<td>4.8</td>
<td>69</td>
<td>6.9</td>
<td>4.3</td>
</tr>
<tr>
<td>5.6 to 8.0</td>
<td>2.4</td>
<td>6.8</td>
<td>134</td>
<td>13.4</td>
<td>5.6</td>
</tr>
<tr>
<td>8.0 to 11.2</td>
<td>3.2</td>
<td>9.6</td>
<td>249</td>
<td>24.9</td>
<td>7.8</td>
</tr>
<tr>
<td>11.2 to 16.0</td>
<td>4.8</td>
<td>13.6</td>
<td>259</td>
<td>25.9</td>
<td>5.4</td>
</tr>
<tr>
<td>16.0 to 22.4</td>
<td>6.4</td>
<td>19.2</td>
<td>160</td>
<td>16.0</td>
<td>2.5</td>
</tr>
<tr>
<td>22.4 to 32.0</td>
<td>9.6</td>
<td>27.2</td>
<td>73</td>
<td>7.3</td>
<td>0.8</td>
</tr>
<tr>
<td>32.0 to 44.8</td>
<td>12.8</td>
<td>38.4</td>
<td>21</td>
<td>2.1</td>
<td>0.2</td>
</tr>
<tr>
<td>44.8 to 64.0</td>
<td>19.2</td>
<td>54.4</td>
<td>6</td>
<td>0.6</td>
<td>0.0</td>
</tr>
<tr>
<td>64.0 to 89.6</td>
<td>25.6</td>
<td>76.8</td>
<td>2</td>
<td>0.2</td>
<td>—</td>
</tr>
</tbody>
</table>

\( \sum = 1000 \)

Consider an analysis of a sub-micron powder. For an arithmetic progression of sizes, let the intervals be 2.5 to 7.5, 7.5 to 12.5, and so on, to 67.5 to 72.5 \( \mu \)m. The resolution will then vary from 1 to 0.071 as the particle size increases. A geometric progression with the same number of size intervals is 0.14 to 1.18, 1.18 to 1.68, 1.68 to 2.36, and so on, to 53.7 to 75.5 with geometric means of 1, \( \sqrt{2} \), \( 2 \sqrt{2} \), 4 to 64. The resolution for each size range is constant at 0.34. If there is a constant error in defining the class intervals, say, a 1 \( \mu \)m undersizing, the effect of this error will be dependent on the size with an arithmetic progression being greater for small particles, whereas with a geometric size interval, the effect is independent of particle size.

Three methods of presenting the histogram are available. In the first, a rectangle is constructed over each class interval, the height of which is proportional to the number of particles in that interval (figure 4.8).

![Number-frequency histogram](image)

A far more useful way is to construct rectangles whose areas are proportional to the numbers of particles in the intervals. The total area under the histogram is equal to the number of particles counted, and it is useful to reduce this number to 100 by making the areas under the rectangles equal to the percentages of particles in the intervals so that histograms may be compared irrespective of the number of particles counted (figure 4.9).

If a sufficient number of particles has been counted, a smooth curve may be drawn through the histogram to give a frequency distribution. It is usual to have more than twelve intervals for this reason, with an upper limit of about twenty in order that the number of particles in each interval remains high and the work involved does not become too great.
The mean size in this case is the geometric mean given by:
\[
\log x_F \sum \frac{d\phi}{d \log x} \cdot d \log x = \sum \log x \frac{d\phi}{d \log x} \cdot d \log x
\]
\[
\log x_F = \frac{\sum \log x \cdot d\phi}{\sum d\phi}
\]
(4.48)

(cf. equation (4.40)). \(\phi\) is a general term for the variables \(W, S\) and \(N\), i.e. weight, surface and number.

4.8 Devices for representing the cumulative distribution curve as a straight line

4.8.1 Arithmetic normal distributions

It is common practice to plot size distribution data in such a way that a straight line results, with all the advantages that follow from such a reduction. This can be done if the distribution fits a standard law, such as the normal law. This distribution occurs when the measured value of some property of a system is determined by a large number of small effects, each of which may or may not operate. If a large number of the measurements of the value are made, and the results plotted as a frequency distribution the well-known, bell-shaped curve results.

Although it might be expected that this type of distribution would be common, it seems to occur only for narrow size ranges of classified material. Actual distributions are skewed, usually to the right.

The equation representing the normal distribution is:
\[
y = \frac{1}{\sigma \sqrt{2\pi}} \exp \left[ - \frac{(x - \bar{x})^2}{2\sigma^2} \right]
\]
(4.49)

where \(y = \frac{d\phi}{dx} = f(x)\)

and \(\int_{-\infty}^{\infty} f(x) \, dx = 1\) (i.e. the distribution is normalized),

\(\sigma\) is the standard deviation (\(\sigma^2\) is the variance), \(\bar{x}\) is the mean size and \(\phi\) is a general term for the frequency, being number, length, surface or volume.

Let \(t = \frac{x - \bar{x}}{\sigma}\)

then \(\sigma \, dt = dx\).

Equation (4.49) becomes:
\[
\frac{d\phi}{dr} = \frac{1}{\sqrt{2\pi}} \exp \left( -\frac{t^2}{2} \right)
\]
Particle Size Measurement

Hence: \[
\int_0^\infty \frac{1}{\sqrt{2\pi}} \int_0^t \exp \left( -\frac{r^2}{2} \right) \, dr
\] (4.50)

A plot of \( \frac{d\phi}{dt} \) against \( t \) results in the well-known 'dumb-bell' shape of the normal probability curve (figure 4.11).

![Graph of the normal probability curve.](image)

**Fig. 4.11** The normal probability curve. Relative frequency against standard deviation [68.26% of the distribution lies within 1 standard deviation - 1<\( t <+1 \) of the mean].

Equation (4.50) is the basis for arithmetic probability graph paper and the integral is tabulated in books on statistics (Table 4.14).

| Table 4.14 Integration of the normal probability equation |
|-----------------|---------|
| \( t \)         | Integral |
| 0               | 0.5000  |
| 0.5             | 0.6915  |
| 1.0             | 0.8413  |
| 1.5             | 0.9332  |
| 2               | 0.9772  |
| 3               | 0.9987  |
| 4               | 0.99997 |

The integral from Table 4.14, for \( t = 1 \) is 84.13%, or for \( t = -1 \) is 15.8%; therefore, the standard deviation:

\[
\sigma = \left( \frac{x - \bar{x}}{t} \right) = x_{84.13} - x_{50} = x_{50} - x_{15.87}
\]

A powder whose size distribution fits the normal equation can therefore be represented by two numbers, the mean value and the standard deviation. The fraction of particles lying between given sizes can then be found from the tables giving the areas under the graph between any two ordinates; such tables are published in Herdan's book [38, p.77]. One advantage of this method of plotting is that experimental and operator errors may be smoothed out and the median and standard deviation can be read off the graph. In the illustrated example (figure 4.12), the median size, i.e. the 50% size, is 35 \( \mu \)m, and the standard deviation is half the difference between the 84% and the 16% sizes (15 \( \mu \)m):

![Graph of normal distribution.](image)

**Fig. 4.12** A normal distribution plotted on normal probability paper.

It can be seen from the graph (figure 4.12) that a 1% unit around 95% probability is of about four times the size range as that around the 50% probability. This tends to aggravate the errors discussed under the theory of compensating errors, hence it is usual to draw a best straight line through the central points.

If all particles greater or smaller than a certain particle size have been removed, the curve becomes asymptotic towards these sizes. It is also possible to determine whether the distributions are homogeneous or heterogeneous since, in the latter case, points of inflection occur. These and other cases are discussed in detail by Irani and Callis [39, p. 47].

### 4.8.2 The log-normal distribution

According to the normal law, it is differences of equal amounts in excess or deficit from a mean value which are equally likely. With the log-normal law, it is ratios of equal amounts which are equally likely. In order to obtain a symmetrical curve of the same shape as the normal curve, it is therefore necessary to plot the relative frequency against log size (see figure 4.10).
The equation of the log-normal distribution is obtained by replacing $x$ with $z = \ln x$, in equation (4.49). Then:

$$y = \frac{1}{\sigma_x \sqrt{2\pi}} \exp \left[ -\frac{(z - \bar{z})^2}{2\sigma_x^2} \right]$$  \hspace{1cm} (4.51)

where $y = \frac{d\phi}{d\ln x}$, $\sigma_x$ is the standard deviation of $z$

and $\bar{z} = \frac{\Sigma z d\phi}{\Sigma d\phi}$ ($\phi = N, S$ or $W$)

$$\bar{z} = \frac{\Sigma z d\phi}{\phi}$$

or $\ln x_g = \frac{\Sigma \ln x d\phi}{\phi}$

Therefore $x_g = \left[\sqrt{\Pi x d\phi}\right]^\phi$ \hspace{1cm} (4.52)

$\Pi x d\phi$ is the product of the group data in which the frequency of particles of size $x$ is $d\phi$, that is, the mean of a log-normal distribution is the geometric mean, i.e. the arithmetic mean of the logarithms:

$$x_g^\phi = x_1^\phi \cdot x_2^\phi \cdot \ldots \cdot x_r^\phi \cdot \ldots \cdot x_n^\phi$$

Since the particle size is plotted on a logarithmic scale, the presentation of data on a log-probability graph is particularly useful when the range of sizes is large.

As before, the median particle size of the data presented on the graph (figure 4.13) is the 50% median size (20 $\mu$m) and this is equal to the geometric mean size $x_g$. The geometric standard deviation is:

$$\log \sigma_g = \log x_{g1} - \log x_{g0}$$

$$= \log x_{g0} - \log x_{g1}$$

or

$$2 \log \sigma_g = \log x_{g84} - \log x_{g16}$$

$$= \log \frac{x_{g84}}{x_{g16}}$$  \hspace{1cm} (4.53)

From figure 4.13

$$\log \sigma_g = \frac{1}{2} \log \frac{40}{10}$$

$$= \log 2$$

Therefore:

$$\sigma_g = 2.$$
Particle Size Measurement

Therefore:

\[
\ln x_{GV} = 3 \ln x_{NV} - 2 \ln x_{NS} = \ln x_{GV} - 2.3 \ln^2 \sigma_g \tag{4.55}
\]

If the initial analysis was a weight analysis, the above equations may be utilized using the conversions:

(a) \[ \ln x_{GS} = \ln x_{GN} + 2 \ln^2 \sigma_g \]

(b) \[ \ln x_{GV} = \ln x_{GN} + 3 \ln^2 \sigma_g \tag{4.56} \]

where \( x_{GN}, x_{GS} \) and \( x_{GV} \) are the number, surface and volume geometric mean diameters.

Table 4.15 Log-normal distribution

<table>
<thead>
<tr>
<th>Particle size range (( \mu m ))</th>
<th>Average size (( x ))</th>
<th>Cumulative % oversize ( \phi )</th>
<th>Percentage in range ( d\phi )</th>
<th>( d\phi / d \log x )</th>
<th>( \log x )</th>
<th>( \log x \ d\phi )</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \sqrt{2} - 2 )</td>
<td>1.68</td>
<td>0.225</td>
<td>0.4</td>
<td>0.4</td>
<td>2.7</td>
<td>0.09</td>
</tr>
<tr>
<td>( 2 - 2\sqrt{2} )</td>
<td>2.38</td>
<td>0.376</td>
<td>3.5</td>
<td>3.1</td>
<td>20.5</td>
<td>1.17</td>
</tr>
<tr>
<td>( 2\sqrt{2} - 4 )</td>
<td>3.26</td>
<td>0.526</td>
<td>14.5</td>
<td>11.0</td>
<td>72.8</td>
<td>5.79</td>
</tr>
<tr>
<td>( 4 - 4\sqrt{2} )</td>
<td>4.76</td>
<td>0.677</td>
<td>36.3</td>
<td>21.8</td>
<td>144.2</td>
<td>14.77</td>
</tr>
<tr>
<td>( 4\sqrt{2} - 8 )</td>
<td>6.72</td>
<td>0.827</td>
<td>63.6</td>
<td>27.3</td>
<td>180.8</td>
<td>22.58</td>
</tr>
<tr>
<td>( 8 - 8\sqrt{2} )</td>
<td>9.52</td>
<td>0.978</td>
<td>85.6</td>
<td>22.0</td>
<td>145.7</td>
<td>21.52</td>
</tr>
<tr>
<td>( 8\sqrt{2} - 16 )</td>
<td>13.40</td>
<td>1.029</td>
<td>95.7</td>
<td>10.1</td>
<td>66.9</td>
<td>10.40</td>
</tr>
<tr>
<td>( 16 - 16\sqrt{2} )</td>
<td>19.00</td>
<td>1.179</td>
<td>95.6</td>
<td>3.9</td>
<td>25.8</td>
<td>4.60</td>
</tr>
<tr>
<td>( 16\sqrt{2} - 32 )</td>
<td>26.90</td>
<td>1.430</td>
<td>100.0</td>
<td>0.4</td>
<td>2.7</td>
<td>0.57</td>
</tr>
</tbody>
</table>

Assuming a weight distribution \( (d\phi = dW = x^3 \ dV) \):

\[
\log x_{GV} = \frac{\Sigma \log x \ d\phi}{d\phi} = 0.815
\]

\( x_{GV} = 6.53 \mu m \)

\( \sigma_g \) may also be obtained from the table, but both these values may be obtained more readily from a graph giving:

\[
x_{GV} = x_{median} = 6.6 \mu m \quad \sigma_g = 1.64 \ (\log \sigma_g = 0.215)
\]

The number distribution will have a median:

\[
\log x_{GN} = \log x_{GV} - 6.9 \log^2 \sigma_g = 0.815 - 6.9 \times 0.215^2
\]

\( x_{GN} = 3.28 \mu m \)

Similarly:

\[
\log x_{GS} = \log x_{GV} - 2.3 \log^2 \sigma_g \]

\( x_{GS} = 5.11 \mu m \)

Mode:

\[
\ln x_m = \ln x_{GV} - \ln^2 \sigma_g
\]

\( x_m = 5.11 \)

In each case the slope of the log-normal line, hence the standard deviation \( \sigma_g \), will be the same; from equations (4.54):

\[
X_{LS} = 3.67
\]

\[
X_{VS} = 4.10
\]

\[
X_{VM} = 4.55
\]

\[
X_{NL} = 3.30
\]

\[
X_{NS} = 3.48
\]

\[
X_{NM} = 3.87
\]

4.8.3 The Rosin-Rammler distribution

For broken coal, a distribution function has been developed which has since been found to apply to many other materials [40]. For example, the particle size distribution of moon dust is found to closely follow a Rosin-Rammler distribution, hence it is assumed that the lunar surface was formed as a result of crushing forces due to impact [78].

Let the size distribution of broken coal be obtained by sieving and let the weight percentage retained on the sieve of aperture \( x \) be denoted by \( R \); a plot of \( R \) against \( x \) gives the cumulative percentage oversize curve.

From the probability considerations the authors obtain:

\[
\frac{dF(x)}{dx} = 100nx^{-n-1} \exp (-bx^n) \tag{4.57}
\]

where \( n \) and \( b \) are constants, \( b \) being a measure of the range of particle size present and \( n \) being characteristic of the substance being analysed. Integrating gives:

\[
R = 100 \exp (-bx^n) \tag{4.58}
\]

This reduces to:

\[
\log \log \frac{100}{R} = \text{constant} + n \log x \tag{4.59}
\]

If \( \log \log 100/R \) is plotted against \( \log x \), a straight line results. The peak of the distribution curve for \( n = 1 \) is at \( 100/e = 36.8 \%), and denoting the mode of the distribution curve by \( x_m \) equation (4.58) gives \( b = 1/x_m \).

The sieve opening for \( R = 36.8 \% \) is used to characterize the degree of comminution of the material, and since the slope of the line on the Rosin-Rammler graph depends on the particle size range, the ratio of \( \tan^{-1} (n) \) and \( x_m \) is a form of variance.
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This treatment is useful for monitoring grinding operations for highly skewed distributions, but should be used with caution since the device of taking logs always reduces scatter, hence taking logs twice is not to be recommended.

4.8.4 Mean particle sizes and specific surface evaluation for Rosin-Rammler distributions

The moment-volume mean diameter is given by:

\[ x_{vm} = \frac{\Sigma x \Delta W}{\Sigma \Delta W} \]  \hspace{1cm} (4.60)

Since \( \Delta W = \Delta F(x) \), defining \( F(x) \) as 100 gives from equations (4.57) and (4.60):

\[ x_{vm} = \frac{1}{100} \int_0^\infty 100n bx^n \exp (-bx^n) \, dx \]

\[ = \frac{1}{n/b} \Gamma \left( \frac{1}{n} + 1 \right) \]

The surface-volume mean diameter may be similarly evaluated as:

\[ x_{sv} = \frac{1}{\sqrt[3]{b} \Gamma (1 - \frac{1}{n})}, \quad n > 1 \]

These can be evaluated from tables of gamma functions for experimental values of \( n \) and the specific surface determined.

4.8.5 Other particle size distribution equations

Various other size distribution functions have been proposed. These are usually in the form of two-parameter (\( b \) and \( n \)) potential distribution functions such as:

(a) Gates-Gaudin-Schumann [60, 61]

\[ \Phi_{GGS} = (bx)^n \] \hspace{1cm} (4.61)

(b) Gaudin-Meloy [62]

\[ \Phi_{GM} = [1 - (1-bx)^n] \] \hspace{1cm} (4.62)

where \( \phi \) is the undersize fraction.

Three- and four-parameter functions with more accuracy have also been proposed [63-65].

4.8.6 Simplification of two-parameter equations

Tarjan [66] converted a two-parameter size distribution function from the form \( \phi = f(x) \) to the form \( \phi = f(x_{0.5}) \) where \( x_{0.5} \) is the median size. This results in an easy-to-handle function with a high degree of correspondence to the more complicated logarithmic function (\( \Phi \)) below [67].

\[ \Phi = \frac{1}{\sqrt{2\pi}} \int_{-\infty}^{\infty} \exp \left( -\frac{u^2}{2} \right) \, du \] \hspace{1cm} (4.64)

Let the parameter \( b \) of equations (4.58), (4.61), (4.62) and (4.63) be expressed in terms of \( x_{0.5} \) when \( \phi = 0.50 \) \( (R = 50 \) in equation (4.55)).

\[ CGS: \quad 0.50 = (bx_{0.5})^n \]
\[ RR: \quad 0.50 = \exp (-bx_{0.5}^2) \]
\[ GM: \quad 0.50 = (1 - bx_{0.5})^n \]

Substituting back for \( b \) gives:

equation (4.58)

\[ \Phi_{RR} = 1 - \exp \left( -\frac{x}{x_{0.5}} \right) \ln 2 \] \hspace{1cm} (4.66)

equation (4.61)

\[ \Phi_{GGS} = 0.5 \left( \frac{x}{x_{0.5}} \right)^n \] \hspace{1cm} (4.66)

equation (4.62)

\[ \Phi_{GM} = 1 - \left[ 1 - \left( \frac{x}{x_{0.5}} \right) (1 - \Phi(0.5)) \right]^n \] \hspace{1cm} (4.67)

4.8.7 Evaluation of non-linear distributions on log-normal paper

A bimodal distribution is detectable on log-probability paper by a change in slope of the line. It is also possible to deduce further features of the distribution. Figure 4.14 shows a bimodal distribution in which the parent distributions do not intersect on a log-probability plot. These distributions are asymptotic to the parent distributions. The geometric means of the parent distributions may be obtained by plotting relative percentage frequency against particle size on log-linear paper (figure 4.15). The area under the two quite distinct curves gives the proportions of the two constituents. From the modes to the 34% levels in areas gives the two standard deviations.

Figure 4.16 shows a bimodal distribution in which the parent distributions intersect on a log-probability plot. These distributions are asymptotic to the parent distributions having the widest size range (i.e. high standard deviation). The point of inflection passes through both distributions. If the separation of means is large, these may be obtained from a plot of relative percentage frequency against particle size on log-linear paper. If the separation of means is small, it is difficult to resolve these distributions (figure 4.17).
Figure 4.18 shows a trimodal distribution. This may also be easily resolved into its component parts if the parent distributions do not intersect on log-probability paper (figure 4.19).

![Graph showing trimodal distribution with parent distributions.]

Fig. 4.18 Trimodal distribution with parent distributions.

Figure 4.19 Relative percentage per log-micrometre of a trimodal distribution with little overlap.

![Graph showing relative percentage per log-micrometre.]

Fig. 4.19 Relative percentage per log-micrometre of a trimodal distribution with little overlap.

4.8.8 Derivation of shape factors from parallel log-normal curves

The two curves on figure 4.20 are analyses of the same powder using gravimetric (X-ray and pipette sedimentation) and volumetric (Coulter) techniques.

![Graph comparing Coulter and X-ray methods.]

Fig. 4.20 Comparison between Coulter and X-ray (x) and pipette methods (•).

Multiplying the diameter by the latter technique by a factor of 1.27 brings the two curves into coincidence, hence:

\[ 1.27 \frac{d_{\text{SRK}}}{d_{\nu}} = d_{\nu} \]

Since:

\[ \frac{d_{\nu}}{d_{\text{SRK}}} = \frac{d_{\nu}}{d_{\nu}} \]

this gives:

\[ \frac{d_{\nu}}{d_{\text{SRK}}} = 1.27; \quad \frac{d_{\nu}}{d_{\nu}} = 1.27^2 \]

and, from equation (4.17):

\[ \psi_{\nu} = 0.384 \]

For a spherical particle, these ratios are unity, increasing with increasing divergence from sphericity.
4.9 The law of compensating errors

In any method of size analysis, it is always possible to assign the wrong size to some of the particles. If this error is without bias, the possibility of assigning too great a size is equally as probable as assigning too small a size. This will modify the extremes of the distribution, but will have little effect on the central region. An illustration with particles of mean size 1, √2, 2, 2√2, 4, 4√2, 8, in which 25% in each size range are placed in the size category below and 25% in the size category above, is shown in figure 4.21. An illustration with bias is also shown with particles having the same size ranges as in the first example, but with 25% in each category being placed in the category above.

<table>
<thead>
<tr>
<th>True distribution</th>
</tr>
</thead>
<tbody>
<tr>
<td>o o o o o o o o o o</td>
</tr>
<tr>
<td>o o o o o o o o o o</td>
</tr>
<tr>
<td>Twenty five percent wrongly placed in the size category above</td>
</tr>
<tr>
<td>o o o o o o o o o o</td>
</tr>
<tr>
<td>o o o o o o o o o o</td>
</tr>
<tr>
<td>Twenty five percent wrongly placed in the size category above and below</td>
</tr>
<tr>
<td>o o o o o o o o o o</td>
</tr>
<tr>
<td>o o o o o o o o o o</td>
</tr>
</tbody>
</table>

Fig. 4.21 Law of compensating variables.

It can be seen that there is the same number of particles in each of the central-size categories, irrespective of whether bias is present or not. If the number distribution is converted into a weight distribution, this is still true, but wrongly assigning a size of 8√2 to 25% by number of the particles in the top-size category gives an apparently coarser distribution. For measurement sizes in arithmetic progression of sizes, the effect is small provided sizing is carried out at 10 or more size intervals and, for a log-normal distribution plotted as a relative frequency curve against the logarithm of particle size, the position of the mode is only slightly affected.

Table 4.16 is for a log-normal distribution having a mean size of 8.6 μm and a standard deviation of 0.320. Wrongly placing 25% of each category by weight in the size category above and 25% in the size category below, gives a mean size of 8.6 μm and a standard deviation of 0.284.

<table>
<thead>
<tr>
<th>Upper size limit (μm)</th>
<th>Mean size (μm)</th>
<th>Cumulative percentage undersize</th>
<th>Percentage in range</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1.2</td>
<td>0</td>
<td>0.3</td>
</tr>
<tr>
<td>√2</td>
<td>1.7</td>
<td>0.3</td>
<td>0.50</td>
</tr>
<tr>
<td>2</td>
<td>2.4</td>
<td>1.4</td>
<td>2.15</td>
</tr>
<tr>
<td>2√2</td>
<td>3.4</td>
<td>5.5</td>
<td>6.85</td>
</tr>
<tr>
<td>4</td>
<td>4.8</td>
<td>15.0</td>
<td>17.38</td>
</tr>
<tr>
<td>4√2</td>
<td>6.8</td>
<td>34.0</td>
<td>35.25</td>
</tr>
<tr>
<td>8</td>
<td>9.6</td>
<td>58.0</td>
<td>57.00</td>
</tr>
<tr>
<td>8√2</td>
<td>13.4</td>
<td>78.0</td>
<td>76.28</td>
</tr>
<tr>
<td>16</td>
<td>19.0</td>
<td>91.0</td>
<td>89.385</td>
</tr>
<tr>
<td>16√2</td>
<td>28.9</td>
<td>97.4</td>
<td>96.30</td>
</tr>
<tr>
<td>32</td>
<td>38.0</td>
<td>99.4</td>
<td>99.03</td>
</tr>
<tr>
<td>32√2</td>
<td>53.8</td>
<td>99.9</td>
<td>99.80</td>
</tr>
<tr>
<td>64</td>
<td>76.0</td>
<td>100</td>
<td>99.98</td>
</tr>
</tbody>
</table>

Figure 4.22 shows a log-normal distribution of geometric mean size 10 μm and geometric standard deviation 2. This distribution is deficient in sub-6 μm particles and is asymptotic to this size. If (x - 6) is taken as the particle size, a straight line results to give the original distribution. A similar sort of plot occurs when there is a deficiency of coarse particles and this may be similarly resolved.

Fig. 4.22 Log-normal distribution with deficiency of sub-6 μm particles.
Figure 4.23 shows Andraasen analysis monitoring a grinding operation. Since, in this case, the new surface created is proportional to grinding time, it is possible to predict future performance. Similarly, less accurately, maximum size is inversely proportional to grinding time (see table below).

<table>
<thead>
<tr>
<th>Grinding time $t$ (hours)</th>
<th>Mean particle size $\bar{x}$ ($\mu$m)</th>
<th>Maximum particle size $x_m$ ($\mu$m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>9</td>
<td>5.3</td>
<td>47.7</td>
</tr>
<tr>
<td>13</td>
<td>4.1</td>
<td>53.3</td>
</tr>
<tr>
<td>15</td>
<td>3.75</td>
<td>56.3</td>
</tr>
<tr>
<td>16</td>
<td>3.42</td>
<td>54.7</td>
</tr>
</tbody>
</table>

Hence, the predicted time to grind to 1 $\mu$m mean size is 55 hours; the predicted time to produce sub-10 $\mu$m particles is 24 hours.

Figure 4.24 gives six analyses from a grinding operation. For samples 4 and 5, the grinding variables have been altered.

### 4.10 Alternative notation for frequency distribution

The notation used in this chapter is widely used but an alternative notation has been developed in Germany [41-47]. Although elegant, the German notation requires some memorization and is probably most suitable for frequent usage and computer application. It is included here, in a shortened form, for the sake of completeness.

#### 4.10.1 Notation

Let the fractional number smaller than size $x$ be:

$$Q_0(x) = \int_{x_{min}}^{x} q_0(x) \, dx$$

or

$$q_0(x) = \frac{dQ_0(x)}{dx}$$

Hence $q_0(x)$ is the fractional number in the size range $x$ to $x + dx$. Further:

$$Q_0(x)_{max} = \int_{x_{min}}^{x_{max}} q_0(x) \, dx = 1$$
4.10.4 Relation between moments

Putting \( t = 0 \) in equation (4.73) and substituting \( q_0(x) \) for \( q_r(x) \) in equation (4.71) gives:

\[
M_{k, r} = \frac{\int_{x_{min}}^{x_{max}} x^k x^r q_0(x) \, dx}{M_{r, 0}}
\]

\[
M_{k, r} = \frac{M_{k+r, 0}}{M_{r, 0}} \tag{4.75}
\]

More generally, substituting \( q_r(x) \) from equation (4.73) in equation (4.71) gives:

\[
M_{k, r} = \frac{M_{k+r-r, 0}}{M_{r-r, 0}} \tag{4.76}
\]

Examples

To determine the surface-volume mean diameter from a number distribution put \( t = 0, r = 2, k = 1 \):

\[
M_{1, 2} = \frac{M_{3, 0}}{M_{2, 0}} = x_{sv}
\]

To determine the surface-volume mean diameter from a surface distribution put \( t = 2, r = 2, k = 1 \):

\[
M_{1, 2} = \frac{M_{1, 2}}{M_{0, 2}}
\]

4.10.5 Means of distributions

(a) Distribution means are given by the equation:

\[
\bar{x}_{1, r} = \frac{\int_{x_{min}}^{x_{max}} x q_r(x) \, dx}{\int_{x_{min}}^{x_{max}} q_r(x) \, dx}
\]

\[
\bar{x}_{1, r} = \frac{M_{1, r}}{M_{0, r}} = \frac{M_{r+1, 0}}{M_{r, 0}}
\]

(b) Arithmetic means are given by the equation:

\[
\bar{x}_{k, 0} = \frac{\int_{x_{min}}^{x_{max}} x^k q_0(x) \, dx}{\int_{x_{min}}^{x_{max}} q_0(x) \, dx}
\]

\[
\bar{x}_{k, 0} = k \sqrt{(M_{k, 0})} \tag{4.78}
\]
4.10.6 Standard deviations

For a number distribution, the variance is defined by:

\[
\sigma_0^2 = \int_{x_{\text{min}}}^{x_{\text{max}}} (x - \overline{x})^2 q_0(x) \, dx
\]

\[
\sigma_0^2 = \int_{x_{\text{min}}}^{x_{\text{max}}} x^2 q_0(x) \, dx - \overline{x}_{\overline{x}}^2 \int_{x_{\text{min}}}^{x_{\text{max}}} q_0(x) \, dx
\]

\[
\sigma_0^2 = M_{2,0} - (M_{1,0})^2
\]

More generally:

\[
\sigma_t^2 = \int_{x_{\text{min}}}^{x_{\text{max}}} (x - \overline{x}_t)^2 q_t(x) \, dx
\]

\[
\sigma_t^2 = M_{2,t} - (M_{1,t})^2
\]

Alternatively:

\[
\sigma_t^2 = M_{1,t} \left[ M_{2,t} - M_{1,t} \right]
\]

Putting \(t = r + 1\) and \(k = 1\) in equation (4.76) gives:

\[
\sigma_t^2 = M_{1,r} \left[ M_{1,r} + M_{1,t} \right]
\]

From equation (4.79):

\[
\sigma_t^2 = \overline{x}_{t+1} - \overline{x}_t
\]

4.10.7 Coefficient of variance

\[
C_t^2 = \frac{\sigma_t^2}{(\overline{x}_t)^2}
\]

\[
= \frac{\overline{x}_{t+1} - \overline{x}_t}{\overline{x}_t} - 1
\]
Particle Size Measurement

hence:

\[ S_v = \frac{\alpha_v}{M_{3,0}} \left[ \frac{M_{2,0}(x_u - x_u)}{M_{3,0}} \right] \times \left[ \frac{1}{Q_3(x_u) - Q_3(x_r)} \right] \]

(4.88)

The application of this equation enables a surface area to be calculated from a summation of increments, i.e.

\[ S_v = \frac{\alpha_v}{M_{3,0}} \sum_{i=1}^{n} M_{2,0}(x_{ei}, x_{ui}) \]

(4.89)

\[ e_i = \min, u_i = \max. \]

4.10.9 Transformation of abscissa

Suppose, in an analysis, \( \xi \), which is a function of \( x \), is measured. Since the amount of material between sizes \( x \) and \( x + dx \) is constant, there must be a simple relationship between \( q(\xi) \) and \( q(x) \). Let

\[ x = f(\xi) \] so that \( \xi = \phi(x) \)

then

\[ q_x(\xi) = q_x(x) \cdot \frac{dx}{d\xi} \]

(4.90)

For example, suppose the following relationship holds:

\[ \xi = x^k \]

then

\[ \frac{d\xi}{dx} = k \xi^{k-1} \]

and

\[ q_x(\xi) = \frac{1}{k \xi^{k-1}} q_x(x) \]

(4.91)

In general, suppose we have the \( q_x(x) \) distribution and wish to find the \( q_x(x^k) \) distribution. Now from equation (4.73):

\[ q_x(x) = \frac{x^{r-u} q_u(x)}{M_{r-u, u}} \]

Substituting in equation (4.91) gives:

\[ q_x^*(\xi) = \frac{x^{r-u} q_u(x)}{M_{r-u, u}} \cdot \frac{1}{k \xi^{k-1}} \]

(4.92)

Example

In a Coulter counter analysis, the pulse height \( V \) is proportional to particle volume, i.e.

\[ V = p^3 x^3 \]

Calculation of \( M_{r,0} \) \([= \bar{x}_r(\xi)]\)

By definition:

\[ M_{r,0} = \int_{x_{min}}^{x_{max}} x^r q_0(x) \, dx \]

\[ = \frac{1}{p^r} \int_{q_{min}}^{q_{max}} \xi^{r+3} q_0(\xi) \, d\xi \]

(4.93)

Also, since \( q_0(x) = x^r q_0(x) \)

\[ \frac{M_{r,0}(\xi)}{p^r} \]

substituting in equations (4.90) and (4.94):

\[ q_x(x) = x^r q_x^*(\xi) \cdot \frac{d\xi}{dx} \cdot \frac{p^r}{M_{r+3,0}(\xi)} \]

\[ q_x(x) = 3px^{r+2} q_x^*(\xi) \]

To calculate \( M_{k,r} \)

\[ M_{k,r} = \int_{x_{min}}^{x_{max}} x^k q_x(x) \, dx \]

\[ = \frac{1}{p^k} \int_{q_{min}}^{q_{max}} \xi^{k+r+3} q_x^*(\xi) \, d\xi \]

\[ = \frac{M_{(k+r)+3,0}(\xi)}{M_{r+3,0}(\xi)} \]

(4.95)
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To calculate volume-specific surface:

\[ S_v = \alpha_{sv} \frac{M_{2,0}}{M_{5,0}} \]

which, using equation (4.95), may be written:

\[ S_v = \alpha_{sv} \cdot p \frac{M_{2,3,0}(\xi)}{M_{1,0}(\xi)} \]  

(4.96)

Thus, specific surface can be determined from moments calculated directly from Coulter counter data.

**Calculation of mean size \( \bar{x}_{k,r} \)**

\[ \bar{x}_{k,r} = k \frac{1}{p} \left( \frac{M_{(k+r)/3,0}(\xi)}{M_{1,0}(\xi)} \right) \]  

(4.97)

4.11 Phi-notation

In geological literature dealing with particle size distribution, a very advantageous transformation of particle size is commonly used. Because it is a logarithmic transformation, it simplifies granulometric computations in the same way as logarithms in mathematical operations. This transformation replaces ratio scale numbers, based on millimetre values of particle size, by interval scale numbers, based on the logarithms of those values. Although several transformations based on decadic logarithms were also suggested (zeta-transformation [55] and gamma-transformation [48]), more than thirty years ago, it has been broadly adopted, particularly in the USA. After the redefinition by McManus [49] and the results of Krumbein [11], the transformation is:

\[ \phi = - \log_{10} \frac{x}{X} \quad \text{or} \quad x = 10^{-\phi} \]  

(4.98)

where \( x \) is a dimensionless ratio of a given particle size, in millimetres, to the standard particle size of 1 mm.

Phi-values can be found if the common decadic logarithms of \( X \) are multiplied by \( \log 10^2 \), i.e. \( x \) values can be converted into their millimetre (or more precisely \( X \)) equivalents if their decadic antilogarithms are multiplied by \( \log 10^2 = 0.30103 \). For easy manipulation, a conversion chart [50, p. 244] or a conversion table [51, 52] can be used.

By using the phi-notation, the statistical measurements acquire a great simplicity. The standard deviation, \( \sigma_\phi \), used in this notation refers to its quartile estimate:

\[ \sigma_\phi = 0.5(\phi_{25} - \phi_{75}) \]  

(4.99)

Similarly the \( \phi \) skewness:

\[ \phi = \frac{\phi_{16} + \phi_{84} - 2\phi_50}{\phi_{84} - \phi_{16}} \]  

(4.100)

Other statistical measurements used in geology for particle size distribution characterization (moment, quartile and others) have been reviewed [53, 54].

4.12 Manipulation of the log-probability equation

Consider the log-normal equation:

\[ \frac{d\phi}{d \ln x} = \frac{1}{\ln \sigma_\phi \sqrt{2\pi}} \exp \left[ - \frac{(\ln x - \ln x^*)^2}{2 \ln \sigma_\phi^2} \right] \]  

(4.101)

\( \phi \) being a general term for the frequency, being number, length, surface or volume (weight) (i.e. \( \phi = N, L, S \) or \( V \)).

Let

\[ x = \frac{(\ln x - \ln x^*)}{\sqrt{2 \ln \sigma_\phi}} \]  

(4.102)

then

\[ \sqrt{2 \ln \sigma_\phi} \cdot dX = d \ln x \]  

(4.103)

and

\[ \int d\phi = \frac{1}{\sqrt{\pi}} \int \exp(-X^2) \cdot dX \]  

(4.104)

The fraction undersize the geometric mean size \( x^* \) is obtained by inserting the limits \( x = 0, x = x^* \), i.e. \( x = -\infty, x = 0 \).

\[ \phi_{x^*} = \frac{1}{\sqrt{\pi}} \int_{-\infty}^{0} \exp(-X^2) \cdot dX \]  

\[ = \frac{1}{2} \]  

(4.105)

Therefore the geometric mean size is the median size.

The fraction lying within one standard deviation of the mean is obtained by inserting the limits \( x = x^*, x = \sigma_\phi x^* \), i.e. \( X = 0, X = 1/\sqrt{2} \).

\[ \phi(x^* - \sigma_\phi x^*) = \frac{1}{\sqrt{\pi}} \int_{0}^{1/\sqrt{2}} \exp(-X^2) \cdot dX \]  

\[ = \frac{1}{\sqrt{\pi}} \left[ X - \frac{X^3}{3} + \frac{X^5}{10} - \frac{X^7}{42} + \ldots \right] * \]  

\[ = 0.3413 \]  

(4.106)

* \[ \exp(-X^2) = \lim_{n \to \infty} \left[ 1 + \left( \frac{-X^2}{n} \right) \right]^n \]  

\[ = 1 + n \left( \frac{-X^2}{n} \right) + \frac{n(n-1)}{2!} \left( \frac{-X^2}{n} \right)^2 + \ldots + \frac{n!}{(n-r)!} \left( \frac{-X^2}{n} \right)^r + \ldots \]  

\[ \int \exp(-X^2) \cdot dX = X - \frac{X^3}{3} + \frac{1}{2!} X^5 - \frac{1}{3!} X^7 + \ldots \]  

\[ + \ldots \cdot \frac{(r-1)!}{(r-1)!} \cdot \frac{X^{2r-1}}{2r-1} \ldots \]
Hence 34.13% of the distribution lies between the median size \( x_\text{g} \) and size \( \sigma_\text{g} x_\text{g} \). (Compare this to the normal-probability curve which contains 34.13% of the distribution between the sizes \( \bar{x} \) and \( \bar{x} + \sigma \).) The geometric standard deviation is therefore: the ratio of the 95.13% and 50% sizes and the ratio of the 50% and 15.87% sizes:

\[
\log \sigma_\text{g} x_\text{g} - \log x_\text{g} = \log x_{84.13} - \log x_{50} = \log x_{84.13/50}
\]

(4.107)

### 4.12.1 Average sizes

Consider a log-normal distribution by number, such that:

\[
\int_{\infty}^{\infty} \frac{dN_r}{r_0} = \frac{1}{\ln \sigma_\text{g} \sqrt{2\pi}} \int_{-\infty}^{+\infty} \exp \left( - \frac{\ln x - \ln x_r}{\sqrt{2 \ln \sigma_\text{g}}} \right)^2 d \ln x
\]

(4.108)

i.e. the distribution is normalized.

\( dN_r \) is the number of particles, in a narrow size range, of mean size \( r \); \( x_0 \) and \( x_\infty \) are the smallest and largest particles present in the distribution. \( x_{84/50} \) is the geometric mean (median) of the number distribution and \( \sigma_\text{g} \) is the geometric standard deviation (which is the same for number, length, surface and weight).

\[
x_{NL} = \frac{\sum_{r=0}^{\infty} x_r dN_r}{\sum_{r=0}^{\infty} dN_r}
\]

(4.109)

\[
x_{2 NS} = \frac{\sum_{r=0}^{\infty} x_r^2 dN_r}{\sum_{r=0}^{\infty} dN_r}
\]

(4.110)

\[
x_{3 NV} = \frac{\sum_{r=0}^{\infty} x_r^3 dN_r}{\sum_{r=0}^{\infty} dN_r}
\]

(4.111)

Substituting from equations (4.102) and (4.103) gives:

\[
x_{NL} = \frac{x_{2N}}{\sqrt{\pi}} \int_{-\infty}^{+\infty} \exp \left( \frac{\ln x - \ln x_{2N}}{\sqrt{2 \ln \sigma_\text{g}}} \right)^2 d \ln x
\]

(4.112)

\[
x_{2 NS} = \frac{x_{3N}}{\sqrt{\pi}} \int_{-\infty}^{+\infty} \exp \left( \frac{2 \ln x - \ln x_{3N}}{\sqrt{2 \ln \sigma_\text{g}}} \right)^2 d \ln x
\]

(4.113)

\[
x_{3 NV} = \frac{x_{4N}}{\sqrt{\pi}} \int_{-\infty}^{+\infty} \exp \left( \frac{3 \ln x - \ln x_{4N}}{\sqrt{2 \ln \sigma_\text{g}}} \right)^2 d \ln x
\]

(4.114)

### Making the transformations:

\[
Y_1 = x - \frac{\sqrt{2}}{2} \ln \sigma_\text{g} \quad \text{in equation (4.112)}
\]

\[
Y_2 = x - \left( \frac{3\sqrt{2}}{2} \right) \ln \sigma_\text{g} \quad \text{in equation (4.113)}
\]

\[
Y_3 = x - \frac{\sqrt{2}}{2} \ln \sigma_\text{g} \quad \text{in equation (4.114)}
\]

(4.115) (4.116)

\[
x_{NL} = \frac{x_{2N}}{\sqrt{\pi}} \exp \left( \frac{1}{2} \ln^2 \sigma_\text{g} \right) \int_{-\infty}^{+\infty} \exp \left( -Y_1^2 \right) dY_1
\]

(4.117)

\[
x_{2 NS} = \frac{x_{3N}}{\sqrt{\pi}} \exp \left( 2 \ln \sigma_\text{g} \right) \int_{-\infty}^{+\infty} \exp \left( -Y_2^2 \right) dY_2
\]

(4.118)

\[
x_{3 NV} = \frac{x_{4N}}{\sqrt{\pi}} \exp \left( \frac{9}{2} \ln \sigma_\text{g} \right) \int_{-\infty}^{+\infty} \exp \left( -Y_3^2 \right) dY_3
\]

(4.119)

The integration yields a value \( 1 = \sqrt{\pi} \) giving:

\[
\ln x_{NL} = \ln x_{2N} + 0.5 \ln^2 \sigma_\text{g}
\]

(4.120)

\[
2 \ln x_{NS} = 2 \ln x_{3N} + 2 \ln^2 \sigma_\text{g}
\]

(4.121)

\[
3 \ln x_{NV} = 3 \ln x_{4N} + 4.5 \ln^2 \sigma_\text{g}
\]

(4.122)

Similarly:

\[
4 \ln x_{NM} = 4 \ln x_{2N} + 8 \ln^2 \sigma_\text{g}
\]

(4.123)

### 4.12.2 Derived average sizes

If the number-size distribution of a particulate system has been determined and found to be log-normal, equations (4.109) to (4.112) may be used to determine other average sizes.

For example, the mean size of a weight distribution is given by:

\[
x_{WM} = \frac{\sum_{r=0}^{\infty} x_r^w dN_r}{\sum_{r=0}^{\infty} x_r^t dN_r}
\]

(4.116)

\[
= \frac{x_{2W}^w}{x_{2N}^w} = \frac{x_{3W}^w}{x_{3N}^w}
\]

(4.117)

Therefore

\[
\ln x_{WM} = 4 \ln x_{NM} - 3 \ln x_{NV}
\]

(4.124)
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Substituting from equations (4.122) and (4.123):
\[
\ln x_{y,M} = \ln x_{gN} + 3.5 \ln^2 \sigma_g
\]  \hspace{1cm} (4.125)

Similarly, the mean size of a surface distribution is given by:
\[
\ln x_{gV} = \ln x_{gN} + 2.5 \ln^2 \sigma_g
\]  \hspace{1cm} (4.126)

Using this equation, the specific surface of the particulate system may be determined since:
\[
S_V = 6/x_{gV}
\]  \hspace{1cm} (4.127)

4.12.3 Transformation of the log-normal distribution by count into one by weight

If a number distribution is log-normal, the weight distribution is also log-normal with the same geometric standard deviation. Using the same treatment as was used to derive equation (4.109) gives, for a weight analysis:
\[
\ln x_{y,M} = \ln x_{gV} + \frac{1}{2} \ln^2 \sigma_g
\]  \hspace{1cm} (4.128)

Comparing with equation (4.114) gives:
\[
\ln x_{gV} = \ln x_{gN} + 3.0 \ln^2 \sigma_g
\]  \hspace{1cm} (4.129)

Since the relations between the number-average sizes and the number-geometric mean are known (equations (4.120) to (4.123)), these can now be expressed as relationships between number-average sizes and the weight (volume) geometric mean \(x_{gV}\):
\[
\ln x_{N,L} = \ln x_{gV} - 2.5 \ln^2 \sigma_g
\]  \hspace{1cm} (4.130)
\[
\ln x_{N,N} = \ln x_{gV} - 2.0 \ln^2 \sigma_g
\]  \hspace{1cm} (4.131)
\[
\ln x_{N,V} = \ln x_{gV} - 1.5 \ln^2 \sigma_g
\]  \hspace{1cm} (4.132)
\[
\ln x_{N,M} = \ln x_{gV} - 1.0 \ln^2 \sigma_g
\]  \hspace{1cm} (4.133)

Other average sizes may be derived from the above, using a similar procedure to that used to derive equations (4.125) and (4.126) to give:
\[
\ln x_{y,V} = \ln x_{gV} - 0.5 \ln^2 \sigma_g
\]  \hspace{1cm} (4.134)

Similarly, for a surface distribution, the equivalent equation to equation (4.129) is:
\[
\ln x_{gS} = \ln x_{gN} + 2.0 \ln^2 \sigma_g
\]  \hspace{1cm} (4.135)

Substituting this relationship into equations (4.130) to (4.133) yields the equivalent relationships relating surface-average sizes with the surface-geometric mean diameter.

4.13 Relationship between median and mode of a log-normal distribution

The log-normal equation may be written:
\[
\frac{d\phi}{dx} = \frac{1}{x \ln \sigma_g \sqrt{2\pi}} \exp\left(-\frac{x^2}{2}\right)
\]

where
\[
2 \ln^2 \sigma_g X^2 = (\ln x - \ln x_m)^2
\]
\[
\sqrt{2 \ln \sigma_g} \frac{dX}{dx} = \frac{1}{x}
\]

At the mode:
\[
\frac{d^2 \phi}{dx^2} = 0 = -\sqrt{2} X - \ln \sigma_g
\]

i.e.
\[
\ln x_m = \ln x_g - \ln^2 \sigma_g
\]  \hspace{1cm} (4.135)

where \(x_m\) is the mode.

4.14 An improved equation and graph paper for log-normal evaluations [56]

Using the relationship:
\[
\frac{1}{x} = \exp(-\ln x)
\]  \hspace{1cm} (4.137)

equation (4.101) may be transformed into the following form:
\[
\frac{d\phi}{dx} = \frac{1}{\ln \sigma_g \sqrt{2\pi}} \exp(-\ln x) \exp\left(-\frac{1}{2 \ln^2 \sigma_g} \left(\frac{x}{x_m}\right)^2\right)
\]

\[
= \frac{1}{\ln \sigma_g \sqrt{2\pi}} \exp\left(-\frac{1}{2 \ln^2 \sigma_g} \left(2 \ln^2 \sigma_g \ln x + (\ln x - \ln x_m)^2\right)\right)
\]

Replacing \(x_g\) by \(x_m\) (equation (4.135)):
\[
\{\} = 2 \ln x_m \ln^2 \sigma_g + \ln^4 \sigma_g + (\ln x - \ln x_m)^2
\]

Therefore
\[
\left\{\right\} = -\ln x_m - \frac{\ln^2 \sigma_g}{2} - \frac{(\ln x - \ln x_m)^2}{2 \ln^2 \sigma_g}
\]

Hence:
\[
\frac{d\phi}{dx} = \frac{1}{x_m \ln \sigma_g \sqrt{2\pi}} \exp\left(-\frac{1}{2 \ln^2 \sigma_g} \right) \exp\left(-\frac{\ln^2 x/x_m}{2 \ln^2 \sigma_g}\right)
\]
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This form of the log-normal equation is more convenient for use since the variable only appears once. It may be simplified further:

\[
\frac{d\phi}{dx} = A \exp\left(-b \ln^2 \frac{x}{x_m}\right)
\]

(4.138)

where

\[
b = \frac{1}{2 \ln^2 \sigma_g}
\]

and

\[
A = \sqrt{\frac{b}{\pi}} \exp\left(-1/4b\right) \frac{x_m}{x_g}
\]

The relationship between geometric mean and mode (equation (4.135)) takes the form:

\[
C = \frac{x_m}{x_g} = \exp\left(-\frac{1}{2b}\right)
\]

This modified form of the log-normal equation simplifies parameter determination from log-probability plots of experimental data. The graph paper may be furnished with additional scales of \(b\) and \(C\) both being determined by drawing a line parallel to the distribution through the pole (0.25 \(\mu m\), 50%).

4.14.1 Applications

Consider a log-normal distribution with a geometric mean

\[x_g = 6.75 \mu m\]

and

\[\sigma_g = \frac{x_{g4\%}}{x_{g}\%} = \frac{11.1}{6.75} = 1.64\]

The mode, according to equation (4.135), is:

\[x_m = 5.27 \mu m\]

making:

\[
\frac{d\phi}{dx} = 0.1344 \exp\left[-2.02 \ln^2 \frac{x}{5.27}\right]
\]

This form is particularly useful when further mathematical computation is envisaged such as for grade efficiency, \(G_c(x)\), calculation since:

\[
G_c(x) = E_T \frac{d\phi_c(x)}{dF(x)}
\]

\[= E_T \frac{d\phi}{dx}\]

where \(d\phi_c/dx\) is the relative frequency of the coarse product, \(d\phi/dx\) is the relative frequency of the feed and \(E_T\) is the total efficiency.

References

5 Sieving

5.1 Introduction

Sieving is an obvious means of classification and it has been used since early Egyptian times for the preparation of foodstuffs. The simplest sieves would be made of some woven material but punched plate sieves are recorded in early Egyptian drawings and by 1556 Agricola is illustrating woven wire sieves [1].

Such sieves were used for powder classification and the inception of test sieving did not arise until sieve aperture sizes were standardized. Standard apertures were first proposed in 1867 by Ritteiger who suggested a √2 progression of sizes based on 75 μm [2]. Modern standards are based on a fourth root of two progression, apart from the French AFNOR series which is based on a tenth root of ten. This range has also been extended downwards but the tolerances are rather liberal which limits their acceptance.

Sieves are often referred to by their mesh size which is the number of wires per linear inch. The American ASTM range is from 400 mesh to 424. The apertures for the 400 mesh are 37 μm hence the wire thickness is 26.5 μm and the percentage open area is 34.

Standards are in the process of modification to match the international ISO series which is based on a root-two progression starting at 45 μm.

Sieve analysis is one of the simplest, most widely used methods of particle size analysis, that covers the approximate size range 20 μm to 125 mm using standard woven wire sieves. Micromesh sieves extend the range down to 5 μm or less and punched plate sieves extend the upper range.

The sieve size $d_A$ is the minimum square aperture through which the particles can pass. Fractionation by sieving is a function of two dimensions only, maximum breadth and maximum thickness for, unless the particles are excessively elongated, the length does not hinder the passage of particles through the sieve apertures (figure 5.1).

This definition only applies to sieves having square apertures.

Sieve analysis results can be highly reproducible even when using different sets of sieves. Although most of the problems encountered in sieving have been known for many years and solutions proposed, reproducibility is rarely achieved in practice due to the failure to take cognisance of these problems [3].