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Guide to particle sizing methods

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Contents

Summary of pages

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Foreword

Publishing information

This British Standard was published by BSI and came into effect on 30 November 2007. It was prepared by Technical Committee LBI/37, Sieves, screens and particle sizing. A list of organizations represented on this committee can be obtained on request to its secretary.

Hazard warnings

The guidance contained in this British Standard is of a general nature. It cannot take account of hazards associated with specific materials which, when subjected to recommended treatments may be dangerous.

Information about this document

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0 Introduction

This British Standard provides simple advice for those wishing to obtain particular information on the size distribution of particles in a sample of particulate material. Various methods of sizing exist which are based on several principles. The advice given is aimed at enabling users to select methods appropriate to their needs.

Particles exist as powders or as suspensions in solid, liquid or gaseous media. In the context of this document particles might also be in the form of liquid droplets or emulsions. Methods are available for sizing them in all these conditions. In some cases it might be desirable, or indeed essential, to examine them in their original condition. In other instances it can be advantageous to change the condition of suspension.

The standard provides a listing of the practical choices available and offers a logical approach to decision making. Principal features of each method are available at a glance in tabulated form.

Important criteria that should be borne in mind when an analysis is discussed and some variables that might influence the choice of approach are illustrated. Although many methods of particle sizing are ingenious, sensitive, reliable and repeatable, no method can be regarded as perfect and compromise is frequently necessary.

1 Scope

This British Standard guide discusses the techniques commonly used for particle sizing, in the context of the physical and chemical properties of the sample on which the information is required, and the purpose for which the information is to be used. This information is primarily relevant to particles in the sub-millimetre range.

2 Normative reference

The following referenced document is indispensable for the application of this document.

BS 2955:1993, *Glossary of terms relating to particle technology*

3 Definitions

For the purposes of this British Standard, the definitions given in BS 2955 apply.

4 General

Particle size is an important determinant in particle behaviour and its measurement can be achieved by many methods. The simplest involve determination of particle dimensions by comparison with a standard scale, calliper gap or two-dimensional figure, such as a circle. Single, two- and three-dimensional measurements can be of value on occasions and their usefulness might be enhanced by being complemented with shape descriptions.

Direct visual comparison of specific particles is not possible for particles the size of which is insufficient for resolution by the naked eye or with the aid of a microscope. To estimate the size of such particles, it is necessary to identify a property of the particle that varies as a function of size, and to determine that property for the particle concerned.

Measurement of a single particle is of limited value, as information is usually required on the size distribution of a population of particles. Obtaining this information by recording the dimensions of individual particles without the use of automation can be tedious and slow. Without automation, individual measurement is rarely the method of choice even with large sized particles.

A comparator method involves the segregation of particles through a mesh of finite size. Those particles that are small enough to pass through the holes in a uniform mesh are separated from those that are retained on the mesh. By use of a range of meshes appropriate to the sizes of the particles present, the population can be fractionated into classes whose contribution to the whole may be estimated by the weight of each fraction.

Detailed information, by assessing the proportions of the population falling within channels defined by a series of size limits or thresholds, can be achieved by other methods. The upper size limit of one channel forms the lower size limit of the next. Such methods depend upon each particle being measured and the distribution is thus derived from a process whereby individual particle data are sorted into size categories.

Some methods depend, not upon the measurement of individual particles, but upon the measurement of a characteristic of the entire particle assembly, for example the scattering of light by a dispersed cloud of particles.

5 Particle size distribution

A particle size distribution can be described as:

- a functional relationship between the quantity of particles and some measure of particle size, or
- a description of the size and frequency of particles in a population (BS 2955:1993, 6 **029)**.

Particle size distributions can be expressed as a fraction, which indicates the proportion of the total population occurring in size channels defined by a continuous range of upper and lower limits. These limits are usually chosen between zero and a magnitude greater than the largest particle present, or an arbitrarily defined maximum.

In particle size distributions the quantity axis may be measured and expressed in terms of number, surface, volume or mass. Significant numerical differences occur when the quantity of particles in a particle size distribution are expressed on different quantity axes (e.g. number, surface, volume or mass).

A single 100 µm particle has the same volume as one thousand 10 µm particles. The volume of sample in a specific size class is related to the number of particles in that size class and by the cube of the particle diameter.

The sizing methods discussed in this British Standard depend upon some prior knowledge of the size and nature of the particles present in the sample to be measured. If such knowledge is not available when the sample is supplied, some might be revealed by microscopy. Both optical and electron microscopy, using calibrated measuring devices, constitute methods of particle sizing, and preliminary examination using a microscope is always recommended, even when it is not chosen as the final method of particle size analysis. Microscopy should also be used as a means to check the effectiveness of any pre-treatment of the sample prior to analysis.

Using appropriate techniques, it is possible to obtain size distributions of populations of particles that are solid, liquid or gaseous. It is necessary that, at the time of analysis, the boundaries of the particles should be distinguishable from the boundaries of the other particles and from the continuous phase in which they are dispersed. Physical and chemical knowledge is required of the particle population under consideration to select a suspending medium that allows this criterion to be met.

A general introduction to the techniques discussed in this standard can be found in [2] and [3].

6 Methods of particle size determination

6.1 General

Particle size analysis methods can be divided into three basic approaches: direct (**6.2**); classification (**6.3**) and indirect (**6.4**).

6.2 Direct methods

These examine and estimate the size of each particle separately and individually. These methods include:

- microscopy;
- electrical sensing zone;
- light blockage; and
- single particle light scattering.

6.3 Classification methods

With these methods, the assembly of particles is separated into size groups based upon a physical parameter of the particle.

Such methods include:

- sieving;
- sedimentation;
- field flow fractionation; and
- hydrodynamic chromatography.

6.4 Indirect methods

These are the methods that utilize a property of the particle. The particles are presented as an ensemble from which a size distribution is inferred from the measured property.

Such methods include:

- laser diffraction;
- dynamic light scattering;
- ultrasound; and
- specific surface.

7 Aggregation

It is important to determine whether the particle size distribution is to be obtained from examination of the fundamental particles or whether to measure them in their aggregated state. For example, a raspberry represents an aggregate of fleshy seeds. Do we wish to determine the size distribution of the fleshy seeds or the berry?

Aggregated particles can be reduced to individual particles by de-aggregation prior to analysis. Conversely, prior treatment can cause individual particles to aggregate into groups which are so stable that they are subsequently measured as individual particles. It is important that the potential for aggregation or de-aggregation occurring in particular samples is assessed. Examination by microscopy can be invaluable for determining changes in the degree of aggregation, even if alternative methods are to be used for the quantitative analysis.

8 Size and shape

The shape of particles is an extremely important factor in measuring particle size as it can influence the perception of size by the methods discussed in this guide.

In sieving, for example, the method discriminates the size of irregular particles according to the second largest dimension of the particle. Shape also influences the sedimentation of particles, causing them to appear smaller by mass than their actual mass.

Difficulties in interpretation of results are compounded when particles in a population to be measured have a range of both size and shape. Microscopic examination can provide an assessment of the shape or range of shapes present, allowing the choice of method or methods adopted to be matched to the predicted behaviour of particles under conditions of measurement.

It is common to express particle size in terms of an equivalent spherical diameter but, because many principles can be adopted in particle size measurement, there are many different ways in which equivalence can be established. Consequently, many types of diameter have been defined, to reflect the chosen equivalent property. These include:

- specific volume diameter;
- Stokes' diameter;
- projected area diameter (two outcomes according to particle orientation);
- sieve size of particle;
- Feret's diameter;
- Martin's diameter; and
- equivalent diffraction diameter

all of which are defined in BS 2955.

9 Sampling and dispersion

9.1 Sampling

As with any type of analysis, it is rarely possible, or even desirable, to perform particle size analysis on an entire bulk particulate material. Consequently it is necessary to create a test sample for the analysis. For the results to be meaningful in relation to the bulk, steps shall be taken to ensure that the test sample is truly representative of the bulk. In the context of particle sizing, this demands that the sample and the bulk have the same particle size distribution. Demonstrating this conclusively is difficult since the bulk cannot usually be analysed. However, there are approaches that offer some reassurance, including:

- following approved sampling procedures (see e.g. ISO 14488[1]);
- analysing replicate test samples, each selected according to the approved procedure;
- comparing them to ascertain the level of accord, or differences (i.e. repeatability and reproducibility).

The quantity of material constituting the test sample can also have a material influence upon the final precision of the result. It shall be clear that if all the particles are identical then any one will represent the whole. As the width of the size distribution increases, more and more test sample is required, if precision in the final result is to be maintained (see [4]).

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[1] In preparation

9.2 Dispersion

9.2.1 General

Dispersion is the separation and distribution of one phase (the dispersed phase) in another (the continuous phase). The physical states (phases) usually measured as particles, constituting the dispersed phase, are liquid, gaseous or solid, and methods exist for measuring each when dispersed in liquid, solid or gas. It is possible to measure gas dispersed as bubbles in liquid or a solid, but not as a gas dispersed in another gas as for such a measurement to take place it is necessary for a sensible stationary boundary to exist between the dispersed phase and the continuous phase.

Facilities for analysing samples vary among methods of particle size analysis. Some methods for analysing solid dispersed phases require sample presentation as a powder dispersed in gas and others as a suspension in a liquid.

NOTE For decision trees showing the steps and decisions to be taken with respect to the type of sample under investigation and the final result required, see Annex A.

Converting from one condition to another is simple but retention of the same particle characteristics throughout the transition can be extremely difficult because in changing the state, changes are also brought about in particle characteristics, including their size. Thus, on drying, as occurs on changing a solid in liquid suspension into a powder, particle agglomeration might occur as a result of pressure and/or sinter bridges (salt bridges). Agglomerates formed in these ways are difficult to re-disperse into a particle dispersion to return them to their previous state. In general, freeze-drying introduces fewer changes than evaporative drying.

Introducing a dry powder into a liquid can also change the distribution in a number of ways. Particles might dissolve. Aggregation and flocculation are possible through the formation of bonding due to Van de Waal's forces, molecular and electrostatic attraction. The creation of a uniform distribution of a particulate solid in a liquid consists of three stages: wetting the surface, de-aggregating clumps and stabilizing the dispersed particles against flocculation or re-aggregation. See BS ISO 14887 for further discussion of this subject.

9.2.2 Wetting

Surface wetting is facilitated by the reduction of the surface tension between the powder and the suspending liquid. This can be assisted by the introduction of wetting agents.

As liquid wets the outer surfaces of a quantity of dry powder, it is drawn through into the interior of the heap by capillary action. Any air trapped within the clump becomes compressed. Wetting, penetration and pressure build-up continue until the pressure of the occluded air balances the capillary force pulling the liquid into the heap. The powder within the occluded area cannot be wetted further unless the clump is broken and the air released. This is often achieved by stirring. Such manipulation is described as de-agglomerating.

9.2.3 De-agglomerating

Some suspensions require the introduction of shear into the system to disperse the powder into the bulk of the liquid. This can be achieved by subjecting the suspension to gentle agitation or by introducing it into an ultrasonic field. Addition of surfactants (avoiding any that reacts with the dispersed powder) can aid dispersion and improve stability. Shear can also lead to fragmentation of particles and care has to be taken to avoid or at least minimize such fragmentation. Again, BS ISO 14887 provides advice on particles in liquid suspensions.

9.2.4 Maintaining particle separation

In a suspension, the balance between forces of attraction and repulsion determine whether the particles move to flocculate or remain separated. Both flocculation and sedimentation lead to non-uniformity in a suspension and thus can create difficulties in sampling and particle size measurement. Flocculation also introduces the possibility of overestimating particle size as a result of aggregates being measured rather than unitary particles. Sedimentation and weak clustering might be minimized or eliminated if adequate stirring or other forms of agitation are continued during the measurement.

Much information can be gained about the relationships among particles in a suspension by examining the suspension, in the condition that it is to be measured in, using a light microscope.

The relationship between solid particles and the liquid in which they are suspended is complex and cannot be discussed fully in this guide. Manufacturers' operating manuals on particle sizing instruments offer advice based on users' experience. More fundamental considerations are discussed in [5] and BS ISO 14887.

In cases where liquid or gas forms the dispersed phase in a liquid or gas continuous phase, it might be difficult to prevent coalescence of the particles constituting the dispersed phase. It is therefore also difficult to maintain the dispersion in the required condition. Because of the unstable nature of such dispersions, it is frequently desirable to employ methods that measure the properties of the ensemble, rather than the characteristics of individual particles.

10 Factors affecting the choice of sizing method

10.1 General

The following important factors should be considered when determining the suitability of sizing methods.

10.2 Purpose of particle size analysis

At the start of a project to create a new material from either a freshly formulated source or a new combination of existing materials, it is difficult to forecast what properties of the particle size distribution will finally become significant.

Particle shape has a very significant influence upon the size and quantity reported, and consideration should be given as to which scientific disciplines will use the particle size distribution data. For example:

- in geology, the silting of river basins and harbours is determined by how particles settle in the water column;
- in drug inhalation studies, the aerodynamic transport properties of airborne particles determine their deposition into the lung.

These examples illustrate the importance of understanding both the physical properties of the material together with that of the purpose of the particle size distribution data, before selecting a sizing method.

10.3 Quantity axis by number, surface or volume

A key factor in the decision as to which particle sizing machine to employ is the quantity of particles in each size class to be reported.

If the task is to polish a hard surface using a polishing compound, one or two large over-sized particles might prove to be disastrous by creating scratches. An ideal particle-sizing machine would be one that does not respond to the normal fine particulate producing the polish but only to the over-sized contaminants; this remains a difficult challenge. However it is clearly the low number of large contaminants that create the scratches. These might be insignificant in terms of their volume or surface.

In the case of a drug quantity, determined either by weight or volume, a distribution by the volume of particles that have a specific size is a major factor in determining both the overall therapeutic effect and in governing the rate of dissolution into the blood stream.

As noted in Clause **5**, a single 100 µm spherical particle has the equivalent volume of one thousand 10 µm particles. This leads to the observations that if it is a number distribution that is needed, a machine that responds to number should be chosen. If distribution by volume is needed, a machine that primarily reports by volume should be used.

Numerical transformation between number and volume or the reverse inevitably results in poor results and is to be discouraged.

10.4 Resolution

If the task is to count the red blood cells, white blood cells and platelets in a sample of blood, a high-resolution size distribution by number is an absolute requirement. The electrical sensing zone technique is ideal for this task. To obtain high resolution, many thousand individual particles have to be examined and such methods are time consuming.

Many materials do not require such exacting measurement. Resolution might remain a key decision factor.

10.5 Relative or absolute size distributions

Absolute size distributions are rarely possible due to most particles being non-spherical to some degree. Most particle size distributions are therefore relative.

Such relative distributions are often all that are required for use by a group of people sharing a common understanding of the subject matter.

However, if the result is likely to be used by other scientific disciplines, or by people who are not familiar with the approximations and limitations imposed by specific measurement techniques, it is important to provide size distributions as close to reality as possible in order to avoid costly misunderstandings.

10.6 Quality control

Some situations demand that samples produced at different times always conform to a specification. Variations from a reference sample can be quantified. These conditions frequently apply in a quality control context in which a product of consistent character is being presented. The ability of a method to make measurements on a wide range of materials is thus of less importance. Frequently the primary need of techniques used in this role is for speed and economy, but precision remains a vital characteristic.

If the results are required for quality control purposes, particle size distributions may be considered as relative measurements, where results from one run can be compared with another, obtained on the same instrument under similar conditions. In such instances the magnitude and direction of change are indicative of changes in the manufacturing process, which can have an impact on the quality of the product. Users define acceptable limits for these variations to ensure that they lie within acceptable limits. In these applications the precision of the measurement is of more interest than the accuracy.

If a consensus about the particle size distribution of a product manufactured at several locations and from several production streams is required, then a more absolute representation of the size distribution is needed.

10.7 Fundamental knowledge

Methods based on principles that can take into account particle properties (such as shape) are most versatile in their application to a range of particle types. In general, methods such as microscopy, in which each particle is examined individually, are favoured for such applications. The acquisition of such detailed information is time consuming and hence a low priority for speed is implied if such methods are to be applied to representative samples of most materials. High speed automated imaging methods improve the speed of obtaining such information.

10.8 Particle properties

If an optical method is selected to measure the particle size distribution, the refractive index (real and imaginary) of both the particle and the suspending media will be required.

The density of the particle can impinge upon the method selected. Sedimentation and dynamic light scattering both have an upper limit to the size density product.

Particle shape also has a major influence, not only upon the size reported, but also in how it affects the sampling methods and particle transport methods if segregation is to be avoided.

10.9 Wanted and unwanted particles

Most of the discussion in this guide focuses on particle size analysis designed to provide a full picture of the distribution of particle sizes in a powder or a suspension. There are some cases, however, where information is required only within a limited range, perhaps because particles within this range are particularly dangerous to a process, or perhaps to test the performance of a filter.

In such cases, a method of analysis appropriate to the critical range might have merit and this consideration might have a strong influence on the selection of the method.

10.10 Shape of particles

Particle shape might influence choice of method directly or indirectly. In the direct sense, some methods can be unsuitable for certain shapes. For example, if needle shaped particles are analysed by sieve, only the needle diameter will be determined, and no information regarding particle length will be obtained.

Consider the case in which ellipsoid or cuboid particles are being sized. Even where the particles have uniform shape, assuming that all dimensions could be measured, it would result in a distribution of particle sizes being reported as they will not all be present in the same orientation.

Gauging methods, such as sieving, separate on the basis of the second smallest particle dimension. If a particle is small enough to pass through the mesh in any orientation, it is of little importance that it has a larger face that would prevent it passing through in another orientation.

Flat, plate-like particles are most likely to lay flat when presented on a microscope slide. If an equivalent spherical diameter is reported from the projected area observed, this is likely to be an over-estimate of the volume of such a particle.

10.11 Single particle or ensemble

Some particle size distributions are unstable and the requirement might be for a “snapshot” of a dynamically changing distribution. In such cases, an accurate assessment can only be made by a method that detects a signal representing the ensemble rather than one that builds a distribution from signals representing single particles. Examples of unstable distributions can be found in liquid and gaseous dispersions but they are particularly typical of emulsions and liquid aerosols, where particles coalesce or break up according to the degree of shear present in the system. Methods of measurement in such cases usually depend upon optical properties such as laser diffraction.

10.12 Size range of particles present

Two aspects of size range are relevant to choosing the appropriate method of analysis. One is the range of sizes present in the sample, as different instruments can be constrained to a limited size range.

The other is that many instruments can only operate over part of their full design range on one pass of the sample, or through one particular sensor. That restricted range is called the instrument's dynamic range. In such cases, the sample might have to be divided into sub-populations with different size ranges, in order for the total size distributions to be measured. This can add an undesirable extra stage into the analysis.

10.13 Dry powders

10.13.1 General

Many products are supplied to end users as dry powders. However, many dry powder products are still re-dispersed into a liquid suspension for particle size analysis. It is considered by such suppliers that the re-dispersion method provides the particle size distribution data that they find most favourable. Advances in dry powder presentation methods and analysis are penetrating some areas to advantage.

The several options available for sizing dry powders are discussed in **10.13.2** to **10.13.4**.

10.13.2 Gauging

Particles with two dimensions smaller than those of an orifice are provided with conditions whereby they may pass through, thus separating them from larger particles provided with the same conditions. A common example of gauging is sieving in which undersize particles pass through a screen or mesh in which holes of uniform size are present.

10.13.3 Microscopic mensuration

The particles are presented to a microscope in such a way that, when magnified, their dimensions can be compared with a calibrated scale or two-dimensional figure.

10.13.4 Laser diffraction units

The units can be fitted with a dry powder feeder such that particles are presented as a dispersed aerosol of powder that is caused to pass through the laser beam and into some form of suction recovery system.

10.14 Suspension of solid particles in a liquid

Suspension might be the natural condition of the sample or it might result from suspending a dry powder into a fluid.

A suspension is a common form of presentation for particle sizing. Additionally, suspension may be used as an intermediate stage in preparing samples for sizing, even though the liquid is absent at the time of sizing. A commonly employed strategy is to create an aerosol from a suspension using an atomizer. The concentration of the suspension is kept low to allow a single solid particle to be in the atomized droplets. The liquid evaporates rapidly leaving an aerosol in separated particles. This technique is often applied to calibrate single particle counters.

10.15 Dispersion of liquid particles in another liquid

A dispersion of this type might be an emulsion. Emulsions present particular problems because they might be characteristically less stable than powders or solid-in-liquid suspensions (see ISO 14488[2]).

10.16 Dispersion of solids or liquids in a gas

Such suspensions are described as aerosols. These might be of droplets or solid particles or particles that remain wet with solvent. The evaporation of droplets or wet particles might cause measurement difficulties with some optical techniques.

10.17 Dispersion of liquid droplets, gas bubbles or solids in a second solid

The choice is restricted to methods that can distinguish boundaries between the phases. Microscopy of thin sections offers one possibility and, if the continuous phase transmits sufficient light, diffraction methods capable of detecting characteristics of ensembles might also be appropriate.

10.18 Choice of condition

In some cases it is possible to exercise a choice as to the condition in which particles are sized. Where this is the case the following considerations are relevant.

- It is frequently convenient to subject only a very small sample of a powder to size analysis; indeed some methods only permit this. In such cases, taking a representative analytical sample presents difficulties.
- The introduction of dry particles into a liquid, will, in some cases, alter the size of the particles through, for example, swelling or dissolving. It might also induce differences in inter-particle relationships, and again these might have the effect of increasing or reducing the apparent size of particles, through aggregation or fragmentation (in extreme cases, biological deterioration might be such that the original particle populations can be replaced by populations of suspended bacteria). Care should always be taken to examine for short- and long-term changes of this type, and to select liquids and conditions in which the sample under scrutiny remains unchanged. Where a suspension is the only means of sizing, and change cannot be avoided, the speed of the analysis might be the most important factor in determining the choice of method.

NOTE There will be occasions when the purpose of particle sizing is to detect the changes in particle size distribution within the suspension with respect to time. In such cases it is important that the interactions of particle and suspending medium are understood and controlled according to the requirements of the analysis.

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[2] In preparation

11 Cost

The cost of particle size measurement depends on the degree of sophistication, and hence cost, of particle-size instrumentation, and the skill required to perform the measurement procedures, which are both very variable. Where users have a requirement for many analyses of the same type, purchase of an appropriate instrument and training of personnel will be necessary.

In considering the cost it is recommended that a cost audit be undertaken. The anticipated number of analyses together with the time taken for each analysis, including machine cleaning and sample preparation, represent operator hours expended. Thus an expensive machine that is both easy to operate and provides a rapid turnaround of analysis might be justified if a sufficient number of analyses are required.

However some users might have need for only a small number of analyses, in which case subcontracting the particle size determinations to a specialist service might be more appropriate.

12 Calibration, traceability, validation and verification

The reliability of measurements made by any device is strengthened if they can be related to recognized standards.

Where a device can be calibrated or verified by the user or by the manufacturer, it should be calibrated using suitable internationally certified reference standards, or secondary standards that are directly traceable to them.

Irrespective of whether the device can be calibrated or verified, its performance should be regularly verified with a control material, which should differ from any calibrator material.

For good laboratory practice, the whole measurement process should also be validated. This demands that objective evidence is established to show that the measurement process consistently produces results to within required specifications, consideration being given to all procedural and analytical variations.

Traceable certified reference material, stage reticules for microscopes and other materials for verification are available for most of the methods described3). Particle size distributions have a size axis and a quantity axis. A reference material or method that provides verification of both axes, and hence the complete distribution, are to be preferred.

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3) Traceable calibrator and control materials are available from, for example, the European Bureau de Reference (BCR), the National Institute for Standards and Technology (NIST) Washington, USA, and LGC, Teddington, UK.

In the light of experience, experts agree primary standards defining important and fundamental units. These are represented, where possible, in a physical form that is then maintained under conditions that permit little or no variation in the value of the primary unit. The standard metre is an example of a fundamental linear standard. Secondary standard units are represented by mathematical functions of fundamental units, for example, volume is represented as the cube of the linear standard. For purpose of scientific communication, and to avoid disputes in trading, it has long been recognized that the greatest value can be derived from using measurements that can be related directly or mathematically to fundamental standards. In this way any units of measurement can be used to express results are independent of the method of measurement. If, however, measurements were restricted to only those that can be directly related in this way, many useful devices for measurement would not exist.

In instruments used for measuring particle size, an assessment is made of particle size distribution by recording values of a property analogous to particle size. This is not ideal but it might be the only method to obtain the particle size data. It is acceptable as long as the limitations can be assessed by recipients of the data. Designers, vendors and users of instruments therefore have a responsibility to state the assumptions that are made in justifying claims that the results obtained with the instruments relate, in the manner claimed, to the fundamental units (or units derived mathematically from fundamental units) in which they are expressed.

The mechanism by which the required relationship is achieved within the instrument is known as calibration, and a statement of the mechanism by which the method of calibration is justified provides traceability.

The efficacy of a calibration procedure is context sensitive. Most methods depend upon the use of spherical materials for calibration and there are few problems likely to arise if test samples comprising only spherical particles are analysed. However, real world samples exclusively comprising spherical particles constitute a small minority of samples examined. The use of spherical particles for calibration has the distinct advantage that the result is capable of a common interpretation. With non-spherical particles used as a calibration material no commonly agreed judgement can be made. Non-spherical particles used for day to day verification might be used to demonstrate that the instrument remains stable in operation without any form of drift or degradation in the size or quantity axis.

13 Particle sizing techniques

The more frequently used particle sizing methods, and the application conditions for which they are appropriate, are summarized in Table 1.

The methods listed in Table 1 represent the most commonly used techniques currently to be found in laboratories.

NOTE Their inclusion does not imply any form of endorsement by BSI.

Some of these techniques, such as dynamic light scattering, have found favour due to the ability to measure predominantly µm particles.

Table 1 **Most commonly used particle sizing techniques**

	Laser diffraction	**Sieving**	**Gravity sedimentation**	**Electrozone sensing**	**Image analysis**	**Dynamic light scattering**
Size range, µm	0.05–2 000	Dry: 40 to mm; Wet: 10 to 45	1.0–100	0.4–1 000	Visible: 0.6–500 Electron: 0.002–10	0.001–3
Dynamic range	1 000:1	100:1	20:1	30:1 for each sensor. Range can be extended by using a series of sensors	10:1 per image	10:1
Principal reported quantity	volume	mass	mass	number and volume	number	intensity weighted distribution
Instrument response	angular light scattering	mass	settling speed	electrical pulse	image size	time dependent light intensity fluctuations
Time required for one completed measurement	1–2 min	Dry: 10–30 min Wet: 1–2 h	6–60 min depending upon size range	1–2 min	10–30 min	5–10 min
Initial training period	1–2 days	1 day	1 day	1–2 days	1 day	1–2 days
Resolution	medium to high	low	medium	high to very high	high	low
Validation method	certified reference materials	verification of aperture size	certified reference materials	certified reference materials	certified graticule	certified reference materials
Suspension media	liquids or gases	air or liquid	liquids	liquids	particles deposited on a substrate	liquids
Dry powder measurements	yes	yes	no	no	yes	no
Droplet sprays	yes	no	no	no	possible	no
Concentration	volume fraction < 0.01 limited by multiple scattering	several grams of sample required	upper volume fraction limited to avoid hindered settling	dilute to avoid coincidence	limited number per image	very dilute
Density limitations	none	none	upper size density limit when settling induces turbulence	upper size density limit for large particles	none	upper size density limit that induces sedimentation
ISO Standard	BS ISO 13320-1	ISO 565 ISO 2591-1 [A)]	BS ISO 13317	BS ISO 13319	BS ISO 13322	ISO 13321 [B)]

[A)] Dual numbered with BS 1796-1.

[B)] Dual numbered with BS 3406-8.

The techniques listed in Table 2 have important properties for certain applications but are employed less frequently. This list is not exhaustive.

Table 2 **Less commonly used particle sizing techniques**

	Hydrodynamic chromatography	**SEM**	**TEM**	**Size exclusion chromatography**	**Ultrasound**
Size range, µm	0.01–3	few nm to 100s µm	sub nm to 10s of µm	0.001–0.1	0.01–1 000
Dynamic range	1000:1	single image: 500:1; multiple images from same sample: 100 000:1	single image: 500:1; multiple images from same sample: 1 000 000:1	1 000:1	1 000:1
Principal reported quantity	distribution on volume basis	distribution by number basis	distribution by number basis	distribution on number basis	distribution on weight basis
Instrument response	separation then light obscuration	contrast	contrast	separation then concentration	attenuation frequency spectra
Time required for one completed measurement	10 min	highly sample dependent (preparation) ~15 min to several hours	highly sample dependent (preparation) ~15 min to several hours	5–40 min	1–15 min
Initial training period	1 day	1–2 days sample preparation. 1 day image analysis	1–2 days sample preparation. 1 day image analysis	1 day	2 days
Resolution	medium to high	very high	very high	medium to high	low to medium
Validation method	certified reference materials	certified test samples, gratings	certified test samples, gratings	certified reference materials	water as reference material
Suspension media	aqueous solution	particles on substrate particles embedded in samples	particles on substrate particles embedded in samples	aqueous solution	liquids
Dry powder measurements	no	yes	yes	no	no
Droplet sprays	no	possible indirectly	possible indirectly	no	no
Concentration	approx. 0.1%	dilute	dilute	approx. 0.1–0.5%	above 1%
Density limitations	none	none (but particles have to exhibit good contrast to distinguish from substrate/matrix)	none (but particles have to exhibit good contrast to distinguish from substrate/matrix)	none	none
ISO Standard	none known	BS ISO 13322-1	BS ISO 13322-1	none known	BS ISO 20998-1

Table 2 **Less commonly used particle sizing techniques** (*continued*)

	Single particle light blockage	**Single particle light scatter**	**Time of flight**	**Centrifugal sedimentation**	**Electrical mobility**
Size range, µm	0.1–10 (instrument dependent)	0.05–10 (instrument dependent)	0.5–20	0.1–25	0.005–1
Dynamic range	100 000:1	10 000:1	10 000:1	>50:1	1 000:1
Principal reported quantity	mass concentration	number concentration	number concentration	mass	number concentration
Instrument response	light obscuration	light scatter	particle velocity in accelerating flow field	settling speed	electrical mobility
Time required for one completed measurement	seconds	seconds	seconds	5–30 min	2 min (scanning) 0.2 s (multi-sensor)
Initial training period	1 day	1 day	1 day	1 day	1 day
Resolution	low to medium	medium to high	high	medium	medium to high
Validation method	CRM	CRM	CRM	certified reference materials	CRM
Suspension media	gas or liquid	gas or liquid	gas	liquid	gas
Dry powder measurements	yes	yes	yes	no	yes
Droplet sprays	yes	yes	yes, but beware of droplet distortion	no	yes
Concentration	0.001–100 mg/m^{-3}	typically up to 10^4 cm^{-3}	typically up to 10^4 cm^{-3}	low	100–10^5 cm^{-3}
Density limitations	no	no	density limits maximum measurable aerodynamic diameter	upper size density limit when settling induces turbulence	no
ISO Standard	BS ISO 21501-3	under development [A)]	under development	BS ISO 13318	under development [B)]

[A)] Attention is drawn to ISO 21501-1, which is in preparation.

[B)] Attention is drawn to ISO 15900, which is in preparation.

Annex A (informative) Flow diagrams to aid the choice of dispersion technique

Figure A.1 **Powder dispersion procedures**

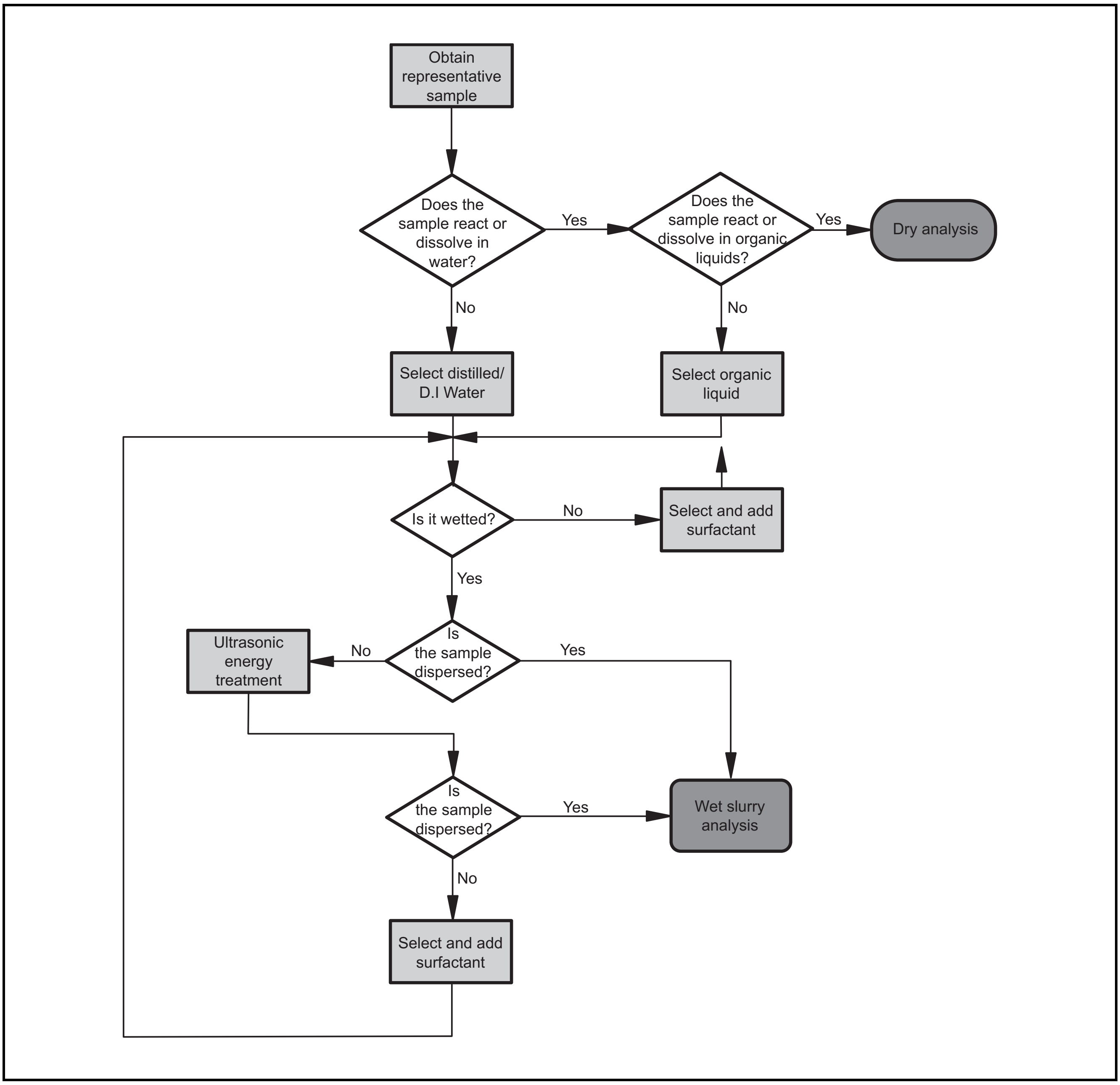
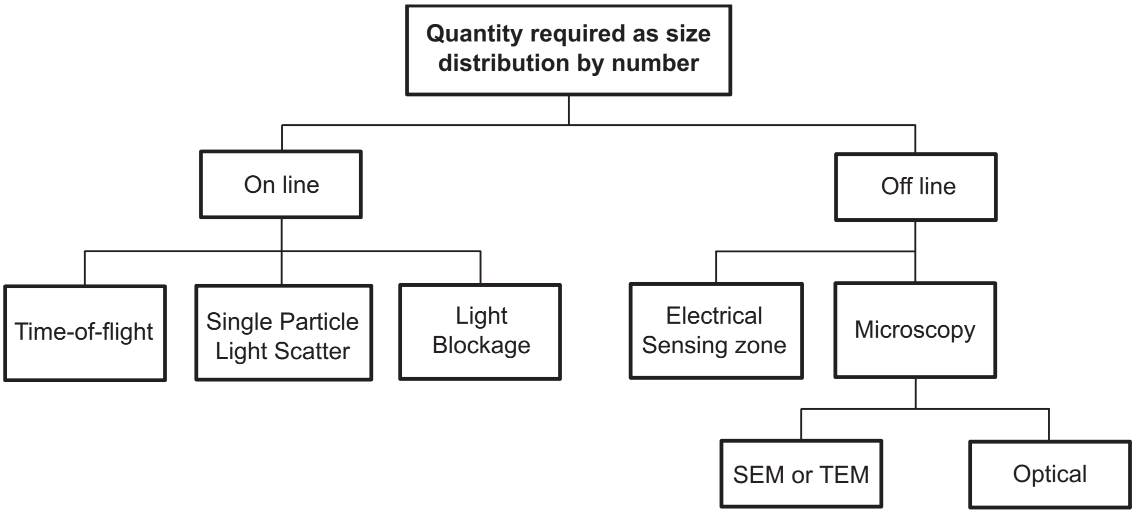
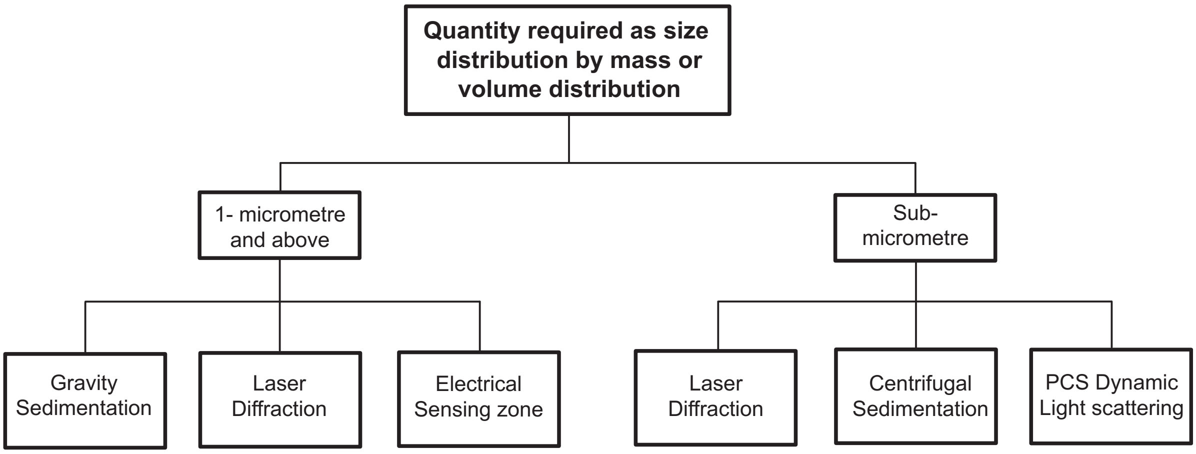


Figure A.2 **Decision trees**

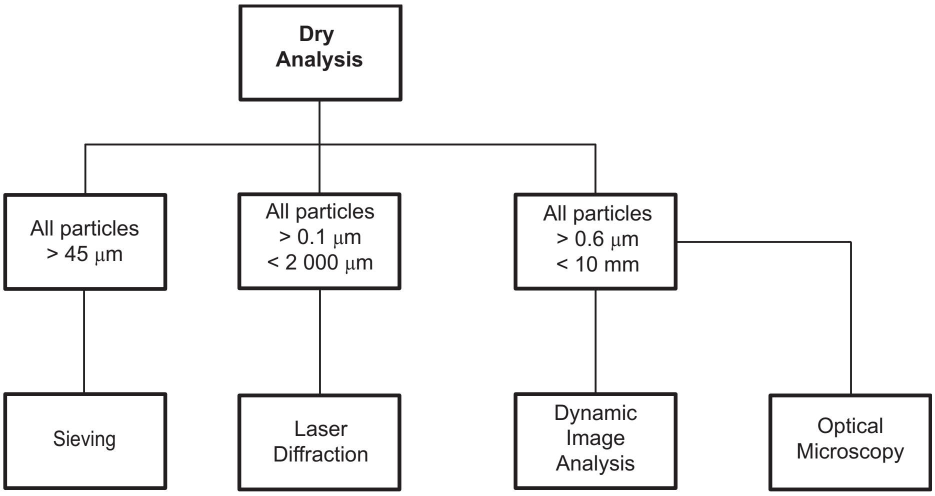
a) Techniques that result in a distribution by number



b) Techniques that result in a distribution by volume



c) Techniques applicable for dry analysis



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