
British Standard Methods for

Determination of particle size distribution

Part 7. Recommendations for single particle light interaction methods

Méthodes de détermination de la granulométrie

Partie 7. Recommandations relatives aux méthodes optiques d'analyse granulométrique des particules uniques

Verfahren zur Bestimmung der Korngrößenverteilung

Teil 7. Empfehlungen zur Bestimmung der Korngrößenverteilung am einzelnen Partikel mit Hilfe optischer Verfahren

Foreword

This new Part of BS 3406, Part 7, was prepared under the direction of the General Mechanical Engineering Standards Committee and is one of a series that recommends methods for determining the size distributions of particles. Current Parts of BS 3406, including this standard, are:

- Part 1 Guide to powder sampling
- Part 2 Recommendations for gravitational liquid sedimentation methods for powders and suspensions
- Part 3 Air elutriation methods
- Part 4 Optical microscope method
- Part 5 Recommendations for electrical sensing zone method (the Coulter principle)
- Part 6 Recommendations for centrifugal liquid sedimentation methods for powders and suspensions
- Part 7 Recommendations for single particle light interaction methods

Particle size distribution is an important basic property. Vast quantities of powdered materials are sold or processed and specifications often include average particle size and/or particle size distribution. Even when other bulk properties are specified, compliance with the specification may be at times achieved only by varying the particle size distribution. Particle size analysis is also used extensively to monitor environmental conditions and in numerous areas of scientific research. In all these contexts the reproducibility and comparability of results is of paramount importance and the use of standardized procedures is essential.

Representative sampling and correct dispersion of particulate material are prerequisites for the determination of size distribution. The procedures described in BS 3406 : Part 1 have been selected to give test portions representative of the bulk.

The techniques used to measure particle size distribution are many and are varied both in principle and in their degree of sophistication and automation. In principle, any property that depends on particle size can be used to define particle size. In practice, the methods that are widely used are both experimentally convenient and based upon a physical principle having a well defined relationship to particle size.

The choice of the most suitable method will depend on the following considerations:

- (a) the purpose for which the analysis is required, e.g. quality control, research, specification requirements;
- (b) wherever possible, the dimension measured should relate to the purpose for which it is required, e.g. Stokes diameter for inertial separation;
- (c) the size range and other properties of the particles, e.g. density, solubility, refractive index;
- (d) aggregation and dispersion characteristics of the particulate matter;
- (e) the amount of material available for analysis;
- (f) the method by which the particulate matter has been sampled;

(g) the conditions under which the particulate matter will be used, for example the state of dispersion in the test should be related to the state of dispersion in the application;

(h) resources available.

The microscope method is the only method in which direct observation is made of the particles. Most methods of particle size measurement ascribe a single dimension to each particle, usually expressed as the diameter of the spherical particle equivalent to some aspect of its properties, such as volume, surface, settling velocity or light scattering. Physical properties of the particles, such as solubility, friability, refractive index and conductivity, preclude the use of some methods. Nevertheless the same particles can be analysed using a range of techniques and if different size parameters are determined different size distributions will result. The intercomparison of results can only be reliable if each laboratory is using a representative sample and is measuring the same parameter by reliable equipment and procedures. Standardization between laboratories is best established by the use of certified reference materials, for example those of the Community Bureau of Reference (BCR).

Single particle light interaction methods are widely used to define the concentration and size distribution of particles suspended in various fluids. Particles can be counted and sized rapidly. In the case of light extinction, the projected area of each particle in a suspension sample is measured and presented as the diameter of a sphere giving the same projected area. In the case of light scattering the diameter presented is that of a sphere giving equal scattered light intensity. Response is affected by the size of the particle and the optical properties of the particle and surrounding medium; the latter effects can be minimized by choice of optical geometry or by suitable calibration and data processing methods. A limitation to all single particle measuring systems is that the particle concentration must be low enough to minimize particle coincidence in the view volume and electronic saturation effects.

The numbers in square brackets used throughout the text of this standard refer to the bibliographic references given in appendix D.

It has been assumed in the drafting of this British Standard that the execution of its provisions is entrusted to appropriately qualified and experienced people.

WARNING. This British Standard calls for the use of substances and/or procedures that may be injurious to health if adequate precautions are not taken. It refers only to technical suitability and does not absolve the user from legal obligations relating to health and safety at any stage. Attention is drawn to the specific hazards described in clause 3.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

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Recommendations

0 Introduction

Two single particle light interaction methods are discussed in this standard. Both depend on particles being suspended in a transparent fluid of refractive index different from that of the particles, and being detected as they pass one at a time through an illuminated volume of defined dimension. The amount of light produced in the detection system by scattering (light scattering), or the amount of light removed from the direct beam by the combination of scattering out of the beam and absorption by the particle (light extinction) is measured as an electrical pulse and related to the size of the particle.

For particles below 2 μm equivalent spherical diameter, light extinction systems usually create a smaller signal than light scattering systems, but light scattering is more affected by surface characteristics of the particle and the refractive index of both the particle and the medium. Because of physical limitations of the light extinction principle, only light scattering is typically used for the measurement of particles below 1 μm .

1 Scope

This Part of BS 3406 gives recommendations on methods of determination of particle size distribution and concentration by single particle counting and sizing instruments using light interaction effects. This Part also provides information to assist in making comparisons between particle size measurements made in different laboratories and/or by different instruments.

It does not cover condensation nuclei counters or methods which obtain light scattering information coming from a cloud or ensemble of particles simultaneously, for instance light diffraction or photon correlation spectroscopy.

Specialized techniques applicable to study of cell biology or haematology are outside the scope of this Part of BS 3406.

NOTE. The titles of the publications referred to in this standard are listed on the inside back cover.

2 Definitions

For the purposes of this Part of BS 3406 the following definitions apply.

2.1 single particle light interaction. The change of light intensity by a single particle causing light scatter and absorption.

2.2 light scattering. The deviation of light as a result of combined reflection, diffraction and refraction of the direct light beam caused by the particle.

2.3 view volume. The illuminated volume within the sensor.

2.4 coincidence. The presence of more than one particle within the view volume at the same time.

2.5 dynamic range. The equivalent spherical diameter range over which the instrument in use can measure particle size with good resolution.

2.6 equivalent spherical diameter. The diameter ascertained by the particle counting/sizing device in use. It is the diameter of a sphere of the same optical response as that of the particle being measured.

2.7 light extinction. The reduction in intensity of a light beam passing through the view volume caused by the absorption and scattering of the light by a particle.

2.8 saturation level. The pulse counting rate at which the electronic circuitry begins to miscount.

2.9 open cavity laser system. An optical system in which the view volume is between the laser mirrors and is thus subjected to much higher light intensity than is otherwise projected from a laser (see appendix A).

2.10 'monosized' particles. Particles of a narrow size range, having an approximately Gaussian size distribution and a coefficient of variation by diameter of preferably less than 5 % but not more than 13 %.

2.11 sensor. A device through which a sample to be measured is passed and within which the light interaction with a particle occurs to cause a measurable response.

2.12 noise level. The minimum voltage setting of the detection circuitry below which the spurious signals of electrical noise become significant and are recorded as particles.

2.13 threshold setting. The voltage level at which the particle counter detection circuitry is set in order to identify a particle of specific size.

3 Health and safety

3.1 Electrical

High voltages of up to 10 000 volts can be present internally in some devices and precautions should be taken if the covers are removed.

3.2 Optical

Particular care should be taken where a laser is incorporated in the device. Irrespective of the light source, the direct beam should not be allowed to enter the eye.

3.3 Mechanical

When pressurized sampling systems are used, the manufacturer's defined procedures are to be followed.

3.4 Chemical

Chemicals used in the methods may be harmful, toxic, corrosive or flammable. Good laboratory procedures should be observed in their preparation and use; for instance, some organic solvents should be handled only in a fume cupboard, into which the instrument and/or glassware assembly is fitted.

Care should be taken to ensure chemical compatibility where a diluent is necessary or when changing suspension fluids which might react together.

Care should be taken to ensure that instruments counting gas-borne particles do not exhaust harmful materials into the working atmosphere.

4 Principles and limitations of single particle light interaction

4.1 Light extinction

4.1.1 General operation. Figure 1 is a schematic diagram of a typical particle analyser. Particles are caused to pass through the view volume and interrupt a collimated light beam causing a reduction of the light falling on the photo-detector. This light reduction is translated into an electrical pulse. Pulses, processed by the pulse amplifier, are passed through the threshold circuit for size discrimination and subsequent counting. Changes in the intensity of the unattenuated light beam, caused by degradation of cell windows, fluctuation in light source output, changes in the optical density of the fluid and ageing of the optical components, are compensated by means of a base level control.

Start and stop controls allow the data to be presented as, for example, particle numbers versus size for a given volume of fluid.

4.1.2 Response. Provided that the linear portion of the photodetector response curve is adhered to, the pulse generated by the passage of the particle through the view volume may be expressed in simple terms as being proportional to the projected cross-sectional area of the particle. The size of the particle is expressed as the diameter 'd' of a sphere of the same projected area:

$$d = \sqrt{(4a/\pi)}$$

where

a is the cross-sectional area of the particle as represented by the pulse.

In practice the relationship between the projected area of the particle and the generated pulse is modified by the optical properties of the particles and the fluid. Although this effect is normally considered to be minimal it can be further reduced by calibrating, if practicable, with materials of similar optical characteristics to the particles under test. Since most particles are not spherical it should be remembered that the orientation of the particle within the view volume will affect the amplitude of the generated pulse and hence the size as indicated by the instrument. This difference can be reduced by calibrating with particles of similar shape.

4.1.3 Size limitations. The lower size limit of detection, normally considered to be about 1 µm, is caused by unwanted light scattering effects, and the signal to noise ratio of the device.

While in theory there is no upper size limit, a typical figure is 2.5 mm.

4.1.4 Operational limitations

4.1.4.1 General. The accuracy and resolution of the sensor is affected by fluid flow rate and coincidence. The manufacturer should specify the flow rate and concentration limit for each sensor to allow no more than 2 % coincidence error. It is recommended that the concentration limit for the sensor is verified using the procedure in 6.2.3 but with the material under test.

4.1.4.2 Flow rate effects. The flow rate should be held within the response capabilities of the detection system and close to the rate at which the instrument was calibrated.

4.1.4.3 Coincidence. When particles are randomly distributed in a sample of fluid, there is a probability that more than one particle may be present within the view volume at any one time. Figure 2 shows the effect of increasing particle concentration within a typical view volume.

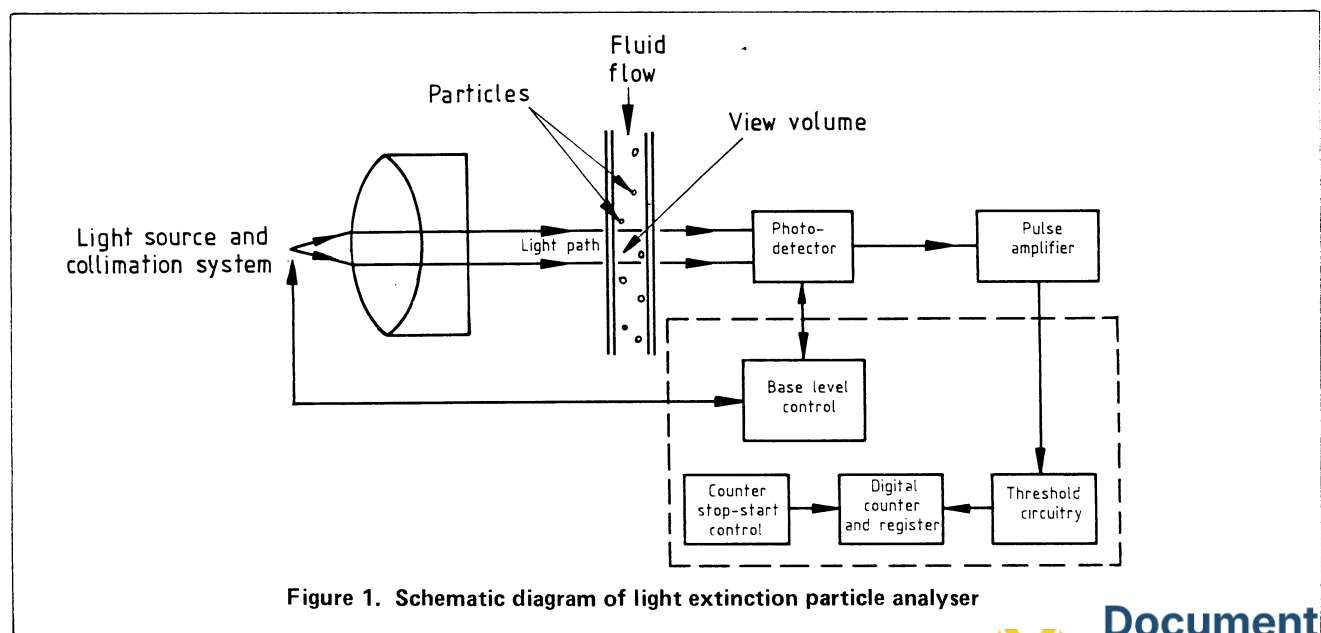


Figure 1. Schematic diagram of light extinction particle analyser

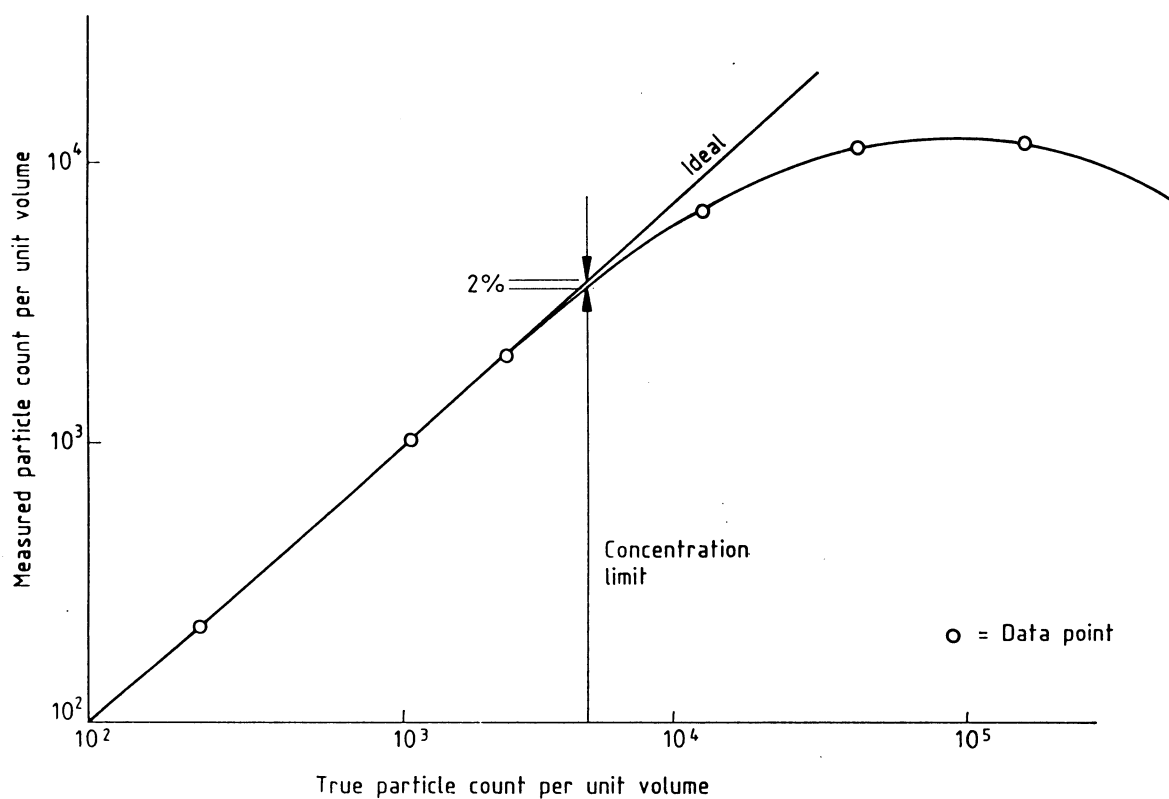


Figure 2. Typical coincidence effect at high concentration

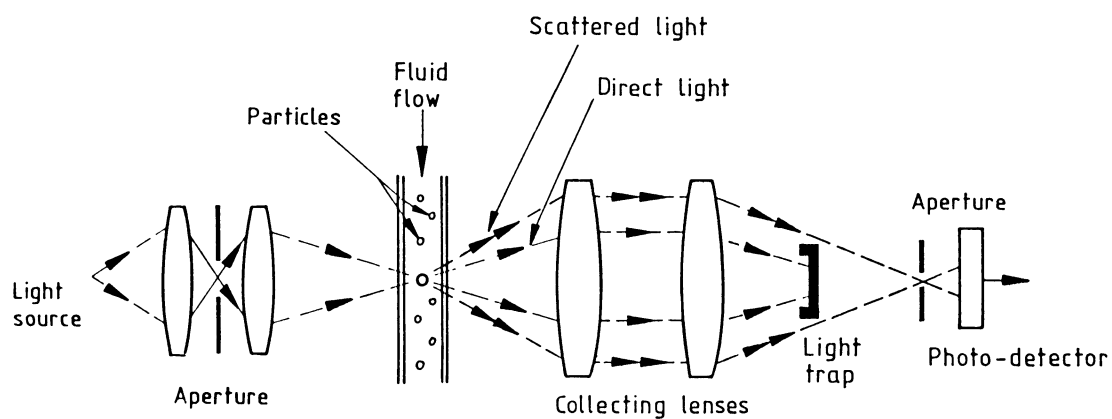


Figure 3. Schematic diagram of near forward scattering optical configuration (from side)

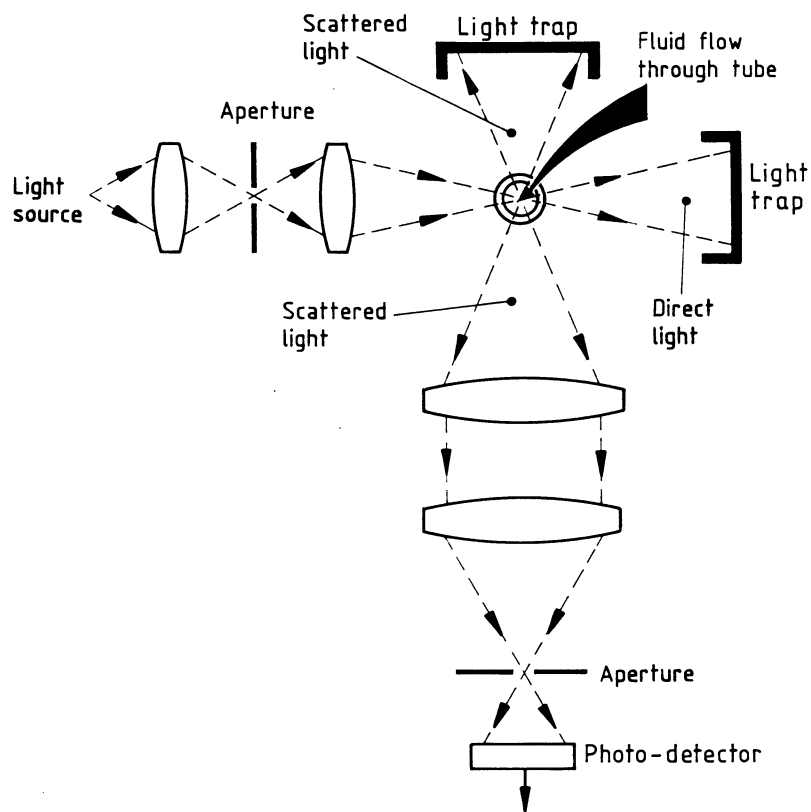


Figure 4. Schematic diagram of right-angle scattering optical configuration

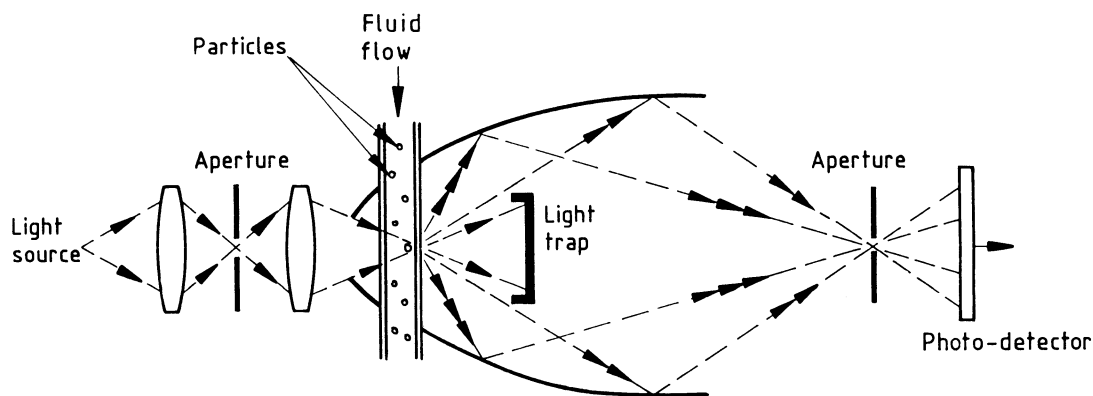


Figure 5. Schematic diagram of wide-angle scattering optical configuration