

Methods for

Determination of particle size distribution —

Part 8: Photon correlation spectroscopy

ICS 19.120

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Committees responsible for this British Standard

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- BLWA Ltd. (The Association of the Laboratory Supply Industry)
- British Fluid Power Association
- China Clay Association
- Institution of Chemical Engineers
- Institution of Electrical Engineers
- Loughborough University of Technology
- Ministry of Defence (Defence Research Agency)
- NABIM
- Royal Pharmaceutical Society of Great Britain
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National foreword

This Part of BS 3406 has been prepared by Subcommittee LBI/37/4. It is identical with ISO 13321:1996, *Particle size analysis — Photon correlation spectroscopy*, published by the International Organization for Standardization (ISO).

ISO 13321 was prepared by Subcommittee 4 of Technical Committee ISO/TC 24, with the active participation and approval of the United Kingdom.

This Part of BS 3406 is one of a series of methods for determining the size distribution of particles. Other Parts of BS 3406 are:

- *Part 1: Guide to powder sampling*;
- *Part 2: Recommendations for gravitational liquid sedimentation methods for powders and suspensions*;
- *Part 3: Air elutriation methods* (obsolescent);
- *Part 4: Guide to microscope and image analysis methods*;
- *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*;
- *Part 9: Recommendations for the filter blockage method (mesh obscuration)*¹⁾.

The particle size distribution of a powder is an important basic property that can distinguish it from other samples of the same composition. 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 achieved 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 techniques is essential.

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

The many techniques used to measure particle size distribution vary both in principle and in degree of technical complexity and automation. In principle, any property that depends on particle size can be used as a means of measuring it. In practice, the methods 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) the size range and other properties of the particles, e.g. density, solubility, refractive index;
- c) aggregation and dispersion characteristics of the powder;
- d) the amount of material available for analysis;
- e) the method by which the sample has been collected;
- f) the conditions under which the powder is to be used, e.g. the state of dispersion;
- g) the resources available.

¹⁾ In preparation.

It has been assumed in the preparation of this Part of BS 3406 that the execution of its provisions is entrusted to appropriately qualified and experienced people. It is expected that users of the standard will be conversant with the appropriate measuring instrument manufacturer's instruction manual.

WARNING. This British Standard may involve 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. In particular, attention is drawn to the Health and Safety Executive Control of Substances Hazardous to Health (COSHH) Regulations (1994).

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

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

Summary of pages

This document comprises a front cover, an inside front cover, pages i to iv, pages 1 to 16, an Inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

Introduction

Particle sizing in the submicrometer size range is nowadays performed on a routine basis using photon correlation spectroscopy (PCS). The success of the technique is mainly based on the facts that it provides estimates of average particle size in measuring times of a few minutes and that user-friendly commercial equipment is available. Nevertheless, proper use of the instrument and interpretation of the results require certain precautions. Therefore there is a need for an International Standard for the determination of particle size by photon correlation spectroscopy, in order to provide a methodology that allows the users to obtain good interlaboratory agreement on accuracy and reproducibility.

Although PCS allows the determination of particle size distribution, this International Standard is limited to the description of size distribution by means of only two parameters: an average size and a polydispersity index, as obtained by so-called cumulants analysis (see annex A). This does not exclude more detailed information about particle size distributions being obtained. However, the reproducibility and reliability of the method of calculation for full distributions is, at the present state of the art, not good enough to include in an International Standard. Again, this does not exclude determination of acceptable size distributions in particular applications.

This International Standard recommends measurements at a single scattering angle of 90° using a He-Ne laser light source with a wavelength *in vacuo* of 632,8 nm. Since solid state laser sources operating at other wavelengths have become available and may be used in future instruments, this International Standard already includes recommendations for such instruments. Although the procedure given is limited to a single angle measurement, with some instruments additional measurements and valuable additional information can be obtained at other scattering angles or by simultaneous analysis of measurements performed at different angles.

This International Standard uses isotropic spherically shaped particles within a test procedure. Measurement of nonspherical and/or nonisotropic particles can be made by this technique where the size of such particles is reported by a spherical equivalence.

A list of suitable references for further reading is given in annex F.

1 Scope

This International Standard describes the application of photon correlation spectroscopy (PCS) to the measurement of an average particle size and a measure of the broadness of the size distribution of particles dispersed in liquids. It is applicable to particle sizes ranging from a few nanometres to about $1\ \mu\text{m}$, or to the onset of sedimentation. In the data analysis procedure (see annexes A and C) it is assumed that the particles are isotropic and spherically shaped.

NOTE 1 The technique is also known or referred to under other names, e.g. quasi-elastic light scattering (QELS) and dynamic light scattering (DLS).

2 Definitions

For the purposes of this International Standard, the following definitions apply.

2.1

average particle diameter, x_{PCS}

harmonic intensity-averaged particle diameter, as determined by equation (C.10) of annex C

it is expressed in nanometres ($10^{-9}\ \text{m}$)

2.2

polydispersity index, PI

dimensionless measure of the broadness of the size distribution, as determined by equation (C.9) of annex C

2.3

scattering volume, V

section of the incident laser beam viewed by the collecting or detector optics

typical order of magnitude is $10^{-6}\ \text{cm}^3$

3 Symbols

B	value of the intercept of the intensity autocorrelation function [see equation (C.6) of annex C];
B_{max}	maximum value of the intercept B for a given setting of the detection optics;
c	concentration of particulate material, in moles per litre;
$G_2(\tau)$	intensity autocorrelation function;
n	refractive index of the dispersion medium;
N_V	number of particles in scattering volume V ;
η	viscosity of the dispersion medium;
Γ	decay rate;
λ_0	laser wavelength <i>in vacuo</i> (632,8 nm for He-Ne laser);

ϕ	particle volume fraction;
ρ	particle density;
θ	scattering angle;
μ_2	second cumulant.

4 Principle

A monochromatic and coherent laser light beam illuminates a representative sample for particle size analysis, dispersed at a suitable concentration in a liquid. The light scattered by the particles at an angle (typically 90°) is recorded by a detector whose output is fed to a correlator. The decay of the autocorrelation function of the scattered intensity is interpreted in terms of average particle size and polydispersity index by the so-called cumulants method.

Annex C provides some theoretical background for particle sizing by PCS.

5 Apparatus

The main components of a typical photon correlation spectrometer are listed below.

NOTE 2 Commercial or “home-made” instruments that meet the requirements of this International Standard may be used. There are several significant differences, both in hardware and software, not only between instruments from different manufacturers but also between different types from one manufacturer. The instrument specifications do not always give adequate information for proper assessment of its specific features. Therefore, annex D has been provided to recommend specifications for PCS instruments.

5.1 Laser, monochromatic, emitting light polarized with its electric field component perpendicular to the plane formed by the incident and detected rays (vertical polarization), e.g. a He-Ne laser capable of 2 mW to 5 mW power output.

5.2 Sample holder, allowing control and measurement of the temperature to within $\pm 0,3$ °C.

5.3 Primary beam stop

5.4 Optics and detector, to collect and digitize the radiation scattered by the sample at an angle of, e.g., 90°. If a polarization analyzer is included, it shall be positioned in the vertical position, i.e. with a maximum transmission for light polarized with its electrical field perpendicular to the plane of incident and scattered beams.

5.5 Correlator

5.6 Computation unit

6 Preliminary procedures

6.1 Instrument location

The instrument shall be placed in a clean environment, free from excessive electrical noise and mechanical vibration and out of direct sunlight. If organic liquids are used (e.g. as an index-matched liquid and/or as the suspension medium), there shall be due regard to local health and safety requirements, and the area shall be well ventilated. The instrument shall be placed on a rigid table or bench to avoid necessity for frequent realignment of the optical system.

NOTE 3 Alternatively, it may incorporate a rigid optical bench internally.

WARNING — PCS instruments are equipped with low or medium power lasers whose radiation can cause permanent eye damage. Never look into the direct path of the laser beam or its reflections. Do not use highly reflecting surfaces when the laser beam is on. Observe local regulations for laser radiation safety.

6.2 Sample preparation and inspection

6.2.1 Samples shall consist of well-dispersed particles in a liquid medium. The dispersion liquid shall fulfil following requirements:

- it shall be transparent (non-absorbing) at the laser wavelength;
- it shall be compatible with the materials used in the instrument;
- it shall not dissolve, swell or coagulate the particulate material;
- it shall have a refractive index different from that of the particulate material;
- its refractive index and viscosity shall be known with an accuracy better than 0,5 %;
- it shall be well filtered.

Water is often used as a dispersion medium. The use of freshly distilled water (the still shall be built from quartz glassware) or of deionized and filtered (pore size 0,2 μm) water is recommended. Since long-range particle interaction may affect the results for strongly charge-stabilized dispersions, a trace of salt [$c(\text{NaCl})$ about 10^{-3} mol/l] may be added to such samples to reduce the range of particle interaction.