INTERNATIONAL STANDARD

ISO 13321

First edition
1996-07-01

Particle size analysis — Photon correlation spectroscopy

Analyse granulométrique — Spectroscopie par corrélation de photons

Reference number
ISO 13321:1996(E)

Contents

International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 13321 was prepared by Technical Committee ISO/TC 24, *Sieves, sieving and other sizing methods*, Subcommittee SC 4, *Sizing by methods other than sieving*.

Annex A forms an integral part of this International Standard. Annexes B to F are for information only.

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.

Particle size analysis — Photon correlation spectroscopy

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 μm, 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} 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} 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 *in vacuo* (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.