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Particle size analysis — Small-angle X-ray scattering

Analyse granulométrique — Diffusion des rayons X aux petits angles

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT), see the following URL: Foreword — Supplementary information.

The committee responsible for this document is ISO/TC 24, *Particle characterization including sieving*, Subcommittee SC 4, *Particle characterization*.

Introduction

This International Standard deals with Small-Angle X-ray Scattering (SAXS), which is performed for particle size analysis in the 1 nm to 100 nm size range. In ideal circumstances, it can provide an estimate of particle size, average size and its distribution, surface area, and sometimes particle shape in a reasonably rapid measurement time. User-friendly commercial instruments are available worldwide from a number of manufacturers for both routine and more sophisticated analyses, and state-of-the-art research instruments are available at synchrotron radiation facilities.

As in all particle size measurement techniques, care is required in all aspects of the use of the instrument, collection of data, and further interpretation. Therefore, there is a need for an International Standard that allows users to obtain good inter-laboratory agreement on the accuracy and reproducibility of the technique.

SAXS can be applied to any hetero-phase system, in which the two or more phases have a different electron density. In most cases, the electron density corresponds reasonably well to the mass density. The so-called 'particle' is always the phase with the smaller volume fraction. Because SAXS is sensitive to the squared electron density difference, it does not matter whether the particles constitute the denser phase and the solvent (or matrix) is the less-dense phase or vice versa. Thus, pore size distributions can be measured with SAXS in the same way as size distributions of oil droplets in emulsions or solid particles in suspensions.

Although SAXS allows the determination of particle size, size distribution, surface area, and sometimes particle shape in concentrated solutions, in powders and in bulk materials, this International Standard is limited to the description of particle sizes in dilute systems. A dilute system in the sense of SAXS means that particle interactions are absent. In case of long range interactions (Coulomb forces between the particles), special care has to be taken and a reduction of the concentration or the addition of salt might be necessary.

Since all illuminated particles present in the X-ray beam are measured simultaneously, SAXS results are ensemble and time averaged across all the particle orientations which are present in the sample.

The shape of the particles can be assigned to a basic geometry: spheroid, disk, or cylinder. This does not exclude more detailed information about the shape of the particle being obtained. However, the method of calculation for more detailed shape analysis is very complex to be included in an International Standard at this time. The sizes of irregularly shaped nanoparticles can be assessed by the radius of gyration (R_g) as obtained by classic Guinier analysis.

The size and size distribution of particles with basic shapes (sphere, disk, cylinder, core-shell, etc.) can be determined from curve fitting for relatively narrow size distributions. The reliability of the method of calculation for broader distributions depends on prior knowledge of the distribution.

This International Standard assumes isotropically oriented nanoparticles of any shape in a test procedure. No dimension of the nanoparticle shall be larger than defined by the scattering accessible to the specific SAXS instrument. This generally limits the largest measureable particle size of the conventional technique to 100 nm, although this limit can be significantly extended in samples with a very narrow size distribution.

Small-angle neutron scattering is not described in this International Standard, but can be used without restriction because the theory and application are similar.

A list of suitable references for further reading is given in the Bibliography.

Particle size analysis — Small-angle X-ray scattering

1 Scope

Small-angle X-ray scattering (SAXS) is a well-established technique that allows structural information to be obtained about inhomogeneities in materials with a characteristic length from 1 nm to 100 nm. Under certain conditions (narrow size distributions, appropriate instrumental configuration, and idealised shape) the limit of 100 nm can be significantly extended. This International Standard specifies a method for the application of SAXS to the estimation of mean particle sizes in dilute dispersions where the interaction between the particles is negligible. This International Standard allows two complementary data evaluation methods to be performed, model fitting and Guinier approximation. The most appropriate evaluation method shall be selected by the analyst and stated clearly in the report. SAXS is sensitive to electron density fluctuations. Therefore, particles in solution and pores in a matrix can be studied in same way.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 26824, *Particle characterization of particulate systems — Vocabulary*

ISO 9276-1, *Representation of results of particle size analysis — Part 1: Graphical representation*

ISO 9276-2, *Representation of results of particle size analysis — Part 2: Calculation of average particle sizes/diameters and moments from particle size distributions*

ISO/TS 27687, *Nanotechnologies — Terminology and definitions for nano-objects — Nanoparticle, nanofibre and nanoplate*

3 Symbols and abbreviations

Table 1 — Symbols

Symbol	Name	Unit
$\bar{d}_{vs}$	Volume-squared-weighted mean particle diameter	nm
$\bar{d}_{num}$	Number-weighted mean particle diameter	nm
I_{out}	Primary beam intensity with sample	
I_{in}	Primary beam intensity without sample	
$I(q)$	Scattered intensity (or scattering intensity)	
q	Momentum transfer or q-value, magnitude of the scattering vector given by $q = (4\pi/\lambda)\sin\theta$	nm^{-1}
r	Particle radius	nm
R_g	Radius of gyration (Guinier radius, see A.4)	nm
t_o	Optimum sample thickness	mm
T	Transmission	