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Particle size analysis — Small-angle X-ray scattering

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National foreword

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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

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.

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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

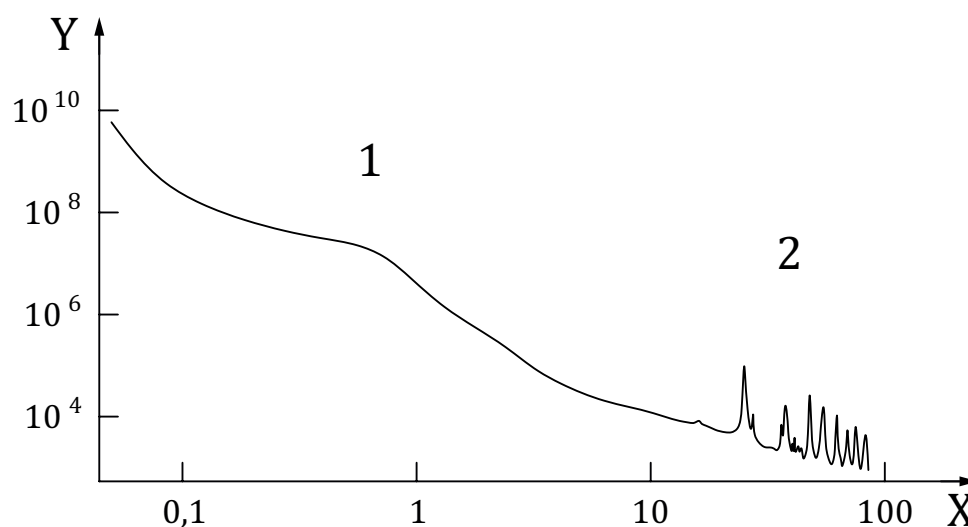
Symbol	Name	Unit
$\bar{d}_{\text{vs}}$	Volume-squared-weighted mean particle diameter	nm
$\bar{d}_{\text{num}}$	Number-weighted mean particle diameter	nm
I_{out}	Primary beam intensity with sample	
I_{in}	Primary beam intensity without sample	
$I(q)$	Scattered intensity (or scattering intensity)	
q	Momentum 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_0	Optimum sample thickness	mm
T	Transmission	

Table 1 (continued)

Symbol	Name	Unit
V	Volume of particle	nm^3
λ	Wavelength of the incident X-rays in vacuum	nm
2θ	Scattering angle	deg or rad
μ	Linear absorption coefficient	mm^{-1}
σ	Standard deviation of size distribution	

4 Principle of the method

When electromagnetic radiation impinges on matter, a small fraction of the radiation is scattered. As a function of the scattering angle or momentum transfer, q -value, the scattered radiation intensity profile contains information that can be used to obtain various characteristics of the material. In particular, when X-rays impinge on a geometrically ordered group of particles or molecules, this gives rise to the well-known X-ray diffraction pattern at wide scattering angles which is used to characterize the unit cell and lattice constants of the material. In the small-angle regime (typically $2\theta < 5^\circ$; wavelength dependent), information on the size of particles or pores within the material is available from the elastic (no change in wavelength) scattering arising from the electron density contrast between the particles and the medium in which they reside. This is analogous to static light scattering. A diagrammatic form of the angular dependence of the X-ray scattered intensity of a titanium dioxide mixture (rutile and anatase) is shown in [Figure 1](#).



Key

- 1 SAXS range
- 2 XRD range
- X scattering angle $2\theta/\text{deg}$
- Y intensity

Figure 1 — X-ray scattering diagram illustrating the small-angle SAXS region (left hand side) and the wide-angle XRD region (right hand side) of a titanium dioxide powder

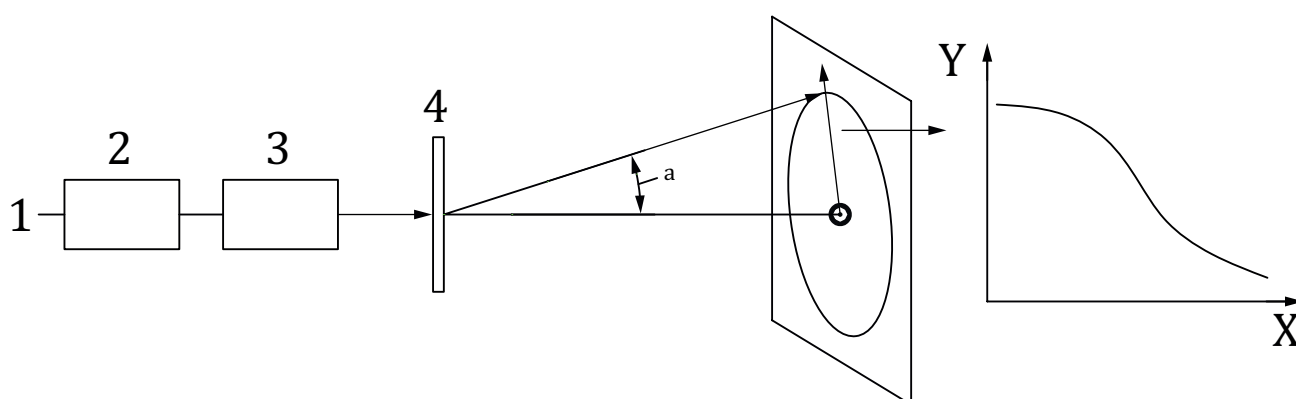
At low concentration, the small-angle scattering region contains information about particle size, size distribution, and particle shape, for which different ranges of q are evaluated. The Guinier approximation

can be applied in the low- q range to get an intensity weighted mean size, when the particles are smaller than $2\pi/q_{\min}$. Model fitting can be applied in the full range of q to compute traceable particle size and size distribution with associated uncertainties. Porod's law can be applied to the high- q range to get an indication of the particle shape. This last method does not provide particle size and is therefore outside the scope of this International Standard. Note that all three methods can fail depending on data quality and particle properties.

At increased concentrations, i.e. those higher than typically one volume %, particle-particle interactions and inter-particle interference can be relevant. Such interactions require sophisticated data modelling and expert knowledge for data interpretation, which is beyond the scope of the present standard. In practice, a concentration ladder may be explored in order to determine the dependence of reported size on concentration. If available, each sample shall be measured twice: in its original concentration, and diluted 1:1 to exclude concentration artefacts. The result of both measurements shall be arithmetically averaged and the uncertainty enhanced by the variation. If dilution is not possible for technical reasons, this shall be stated in the report and the uncertainty shall be marked. Note that the radius of gyration is more affected by concentration than model fitting.

5 Apparatus and procedure

A diagrammatic form of a SAXS instrument is shown in [Figure 2](#).



Key

- 1 X-ray source
- 2 optics
- 3 collimation system
- 4 sample
- a 2θ
- X 2θ or q
- Y scattered intensity

Figure 2 — Diagrammatic form of a SAXS instrument, consisting of X-ray source, optics, collimation system, sample holder, beam stop, and X-ray detector

The SAXS set-up consists of X-ray source, optics, collimation system, sample holder, beam stop, and detector. In order to extract meaningful information from the measurement, the following key parameters define the capability of the system:

- q -range: $q_{\min}$ and $q_{\max}$; number of sampled points in the Guinier region for Guinier approximation;
- detector sensitivity and system background noise.