

# Particle size analysis — Small angle X-ray scattering method

ICS 19.120

## National foreword

This Draft for Development reproduces verbatim ISO/TS 13762:2001

**This publication is not to be regarded as a British Standard.**

It is being issued in the Draft for Development series of publications and is of a provisional nature because it deals with emerging technology, in which insufficient experience has been gained for a full ISO standard to be appropriate. It should be applied on this provisional basis, so that information and experience of its practical application may be obtained.

Comments arising from the use of this Draft for Development are requested so that UK experience can be reported to the international organization responsible for the Technical Specification. A review of this publication will be initiated not later than 3 years after its publication by the international organization so that a decision can be taken on its status at the end of its 3-year life. Notification of the start of the review period will be made in an announcement in the appropriate issue of *Update Standards*.

According to the replies received by the end of the review period, the responsible BSI Committee will decide whether to support the conversion into an international standard, to extend the life of the Technical Specification for another 3 years or to withdraw it. Comments should be sent in writing to the Secretary of BSI Subcommittee LBI/37/4, Sizing by methods other than sieving, at British Standards House, 389 Chiswick High Road, London W4 4AL, giving the document reference and clause number and proposing, where possible, an appropriate revision of the text.

A list of organizations represented on this committee can be obtained on request to its secretary.

**Cross-references**

The British Standards which implement international publications referred to in this document may be found in the *BSI Catalogue* under the section entitled “International Standards Correspondence Index”, or by using the “Search” facility of the *BSI Electronic Catalogue* or of British Standards Online.

This Draft for Development, having been prepared under the direction of the Materials and Chemicals Sector Policy and Strategy Committee, was published under the authority of the Standards Policy and Strategy Committee on 4 December 2002

**Summary of pages**

This document comprises a front cover, an inside front cover, the ISO/TS title page, pages ii to v, a blank page, pages 1 to 22, an inside back cover and a back cover.

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**Amendments issued since publication**

| Amd. No. | Date | Comments |
|----------|------|----------|
|          |      |          |
|          |      |          |
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© BSI 4 December 2002

ISBN 0 580 40601 6

# TECHNICAL SPECIFICATION

# ISO/TS 13762

First edition  
2001-03-15

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## Particle size analysis — Small angle X-ray scattering method

*Analyse granulométrique — Méthode de dispersion par rayons X sous  
angle faible*



Reference number  
ISO/TS 13762:2001(E)

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

The main task of technical committees is to prepare International Standards. 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.

In other circumstances, particularly when there is an urgent market requirement for such documents, a technical committee may decide to publish other types of normative document:

- an ISO Publicly Available Specification (ISO/PAS) represents an agreement between technical experts in an ISO working group and is accepted for publication if it is approved by more than 50 % of the members of the parent committee casting a vote;
- an ISO Technical Specification (ISO/TS) represents an agreement between the members of a technical committee and is accepted for publication if it is approved by 2/3 of the members of the committee casting a vote.

An ISO/PAS or ISO/TS is reviewed every three years with a view to deciding whether it can be transformed into an International Standard.

Attention is drawn to the possibility that some of the elements of this Technical Specification ISO/TS 13762 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO/TS 13762 was prepared by Technical Committee ISO/TC 24, *Sieves, sieving and other sizing methods*, Subcommittee SC 4, *Sizing by methods other than sieving*.

Annexes A and B form a normative part of this Technical Specification. Annexes C to E are for information only.

## Introduction

The size range for which the small angle X-ray scattering (SAXS) method is applicable is approximately in the range of 1 nm to 300 nm. The success of the technique is mainly based on the fact that SAXS effect results from the difference of electron density between particles and their surroundings so that size  $X_{\text{SAXS}}$  always indicates the size of a primary particle rather than the internal crystallite or external agglomerate size; in other words, the requirement of particle dispersion of a sample for SAXS analysis is not as strict as that for other methods.

However, the SAXS method has its limitations: firstly, it cannot distinguish pores from particles; secondly, the interference effect between particles will arise as the sample is available only in concentrated form.

SAXS measurements and the interpretation of the data are currently not uniform. The purpose of this Technical Specification is to facilitate comparisons of size analysis made in different laboratories.

It is well known that X-rays can kill human tissue. However, this Technical Specification does not purport to address all the safety problems associated with the use of the SAXS method; it is the responsibility of the user to establish appropriate safety and health practices prior to its use.



# Particle size analysis — Small angle X-ray scattering method

## 1 Scope

This Technical Specification specifies the method for determining particle size distribution of ultra-fine powders by the small angle X-ray scattering technique. It is applicable to particle sizes ranging from 1 nm to 300 nm. In the data analysis, it is assumed that particles are isotropic and spherically shaped.

The method described in this Technical Specification is also applicable to particle suspensions.

This Technical Specification does not apply to:

- a) powders containing particles whose morphology is far from spherical, except by special agreement;
- b) powders consisting of porous particles;
- c) mixtures of powders.

## 2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this Technical Specification. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this Technical Specification are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

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

ISO 9276-2:—<sup>1)</sup>, *Representation of results of particle size analysis — Part 2: Calculation of average particle sizes/diameters and moments from particle size distributions*.

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1) To be published.

### 3 Symbols and abbreviations

|                  |                                                                                                                                                                             |
|------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| SAXS             | small angle X-ray scattering                                                                                                                                                |
| $x$              | particle size, diameter of a sphere                                                                                                                                         |
| $\Delta x_i$     | length of particle size interval, $\Delta x_i = x_i - x_{i-1}$                                                                                                              |
| $n$              | total number of size classes                                                                                                                                                |
| $\bar{q}_{3,i}$  | average density distribution of the class $\Delta x_i$ by volume or mass                                                                                                    |
| $\bar{q}_{3,i'}$ | average density distribution in a representation with logarithmic abscissa                                                                                                  |
| $Q_{3,i}$        | cumulative volume fraction of particle sizes $x \leq x_i$                                                                                                                   |
| $\varepsilon$    | scattering angle                                                                                                                                                            |
| $\Delta Q_{3,i}$ | volume fraction within the class $\Delta x_i$ , $\Delta Q_{3,i} = \bar{q}_{3,i} \cdot \Delta x_i$                                                                           |
| $\Phi(\zeta)$    | scattering function of a spherical particle:<br>$\Phi(\zeta) = 3(\sin \zeta - \zeta \cos \zeta) / \zeta^3, \quad \zeta = \frac{\pi x}{\lambda} (\varepsilon^2 + t^2)^{1/2}$ |
| $\lambda$        | wavelength of incident X-ray beam                                                                                                                                           |
| $t$              | angle variable along the slit-height direction                                                                                                                              |
| $F(t)$           | slit-weighting function along the slit-height direction                                                                                                                     |

### 4 Principle

As a narrow beam of X-ray passes through a powder layer containing ultra-fine particles, it will be dispersed around the incident beam at a small angle range resulting from the electron scattering in the particles. The distribution of scattered intensity is closely related to the particle size distribution.

The scattered intensity  $I(\varepsilon)$  from a dilute spherical particle system can be expressed by the integral equation:

$$I(\varepsilon) = c \int_{-\infty}^{+\infty} F(t) dt \int_{x_0}^{x_n} \omega(x) x^3 \Phi^2(\zeta) dx \quad (1)$$

where

$x_0$  is the size below which there are no particles;

$x_n$  is the size above which there are no particles;

$\omega(x)$  is the size distribution function by volume (without normalization);

$c$  is a synthetic constant.