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Determination of the specific surface area of porous and particulate systems by small-angle X-ray scattering (SAXS)

Détermination de la surface spécifique pour des systèmes poreux et particulaires par diffusion des rayons X aux petits angles (SAXS)

Reference number
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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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This document was prepared by Technical Committee ISO/TC 24 *Particle characterization including sieving*, Subcommittee SC 4, *Particle characterization*.

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Introduction

Small-angle X-ray scattering (SAXS) can be used to determine the specific surface area of nanoporous (presence of nanopores) and nanoparticulate systems which include mesoporous and partly macroporous materials. SAXS is a well-established method to obtain structural information on inhomogeneities in materials at the nanoscale, typically between 1 nm and 100 nm, and is thus perfectly suited for nanoporous, i.e. materials comprising nanopores and nanoparticulate systems which include mesoporous (presence of mesopores) and partly macroporous (presence of macropores) materials. With special instrumentation, and/or by using absolute-scale techniques, the limits can be significantly extended. 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 measurement techniques for surface area, 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. A 'phase' is in this context understood as a homogeneous electron density domain in the typical size range for SAXS between about 1 nm and 100 nm. State-of-the-art SAXS instruments and synchrotron SAXS beamlines allow significantly extending the limit of 100 nm to several hundred nanometres. Special instrumentation for ultra-small angle X-ray scattering (USAXS) pushes the upper size limit even up to the µm range. This document describes two different evaluation approaches for determining the specific surface area: The Invariant (K/Q) method has an upper size limit for the structure of up to several hundred nanometres, whereas for the absolute-scale method the size of the structure can even be in the µm range.

Because SAXS is sensitive to the squared electron density difference, it does not matter whether the scattering system is composed of pores or particles within a matrix, respectively.

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

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Determination of the specific surface area of porous and particulate systems by small-angle X-ray scattering (SAXS)

1 Scope

This document specifies the application of small-angle X-ray scattering (SAXS) for the determination of specific surface area. Both the mass specific surface area in the order of 1 m^2g^{-1} to 2 000 m^2g^{-1} and the volume specific surface areas in the range from 0,01 m^2cm^{-3} to 1 000 m^2cm^{-3} can be obtained.

The method described is applicable to dilute and concentrated systems.

NOTE In ISO 17867:2020, the determination of the particle size by SAXS is limited to dilute systems.

The determination of surfaces with SAXS is straightforward for two-phase systems only. Surface determination in systems with more than two phases is beyond the scope of this document.

The term 'surface' refers to any interface between domains of different density (more precisely: electron density) and is not restricted to the external surface of particles. As any interfaces between areas with different electron density, not only to air or vacuum, can be probed, the method can be applied to any heterogeneous system.

SAXS measures not only the specific surface area of open pores but also of inaccessible, closed pores or inclusions.

NOTE This is in contrast to gas sorption methods which are described in ISO 9277:2010.

In addition to porous systems, there can be contributions of internal interfaces to the measured specific surface area of any heterogeneous compact solid system, such as between crystalline and amorphous phases, provided there is an electron density contrast. Although materials comprising micropores (pore width < 2 nm) can also be analysed with respect to their specific surface area with SAXS, this document does not cover these materials.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
nanopore
pore with width of 100 nm or less

[SOURCE: ISO 15901-2:2021, 3.10]

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3.2
macropore
pore with width greater than 50 nm

[SOURCE: ISO 15901-1:2016, 3.7]

3.3
mesopore
pore of internal width between 2 nm and 50 nm

[SOURCE: ISO 15901-1:2016, 3.8]

3.4
micropore
pore of internal width less than 2 nm

[SOURCE: ISO 15901-1:2016, 3.9]

3.5
surface area
extent of accessible surface area as determined by a given method under stated conditions

[SOURCE: ISO 15901-1:2016, 3.30]

3.6
mass specific surface area
surface area of the sample divided by sample mass

3.7
volume specific surface area
surface area of the sample divided by sample volume

3.8
external (outer) surface
envelope surface of particles in the micrometre and sub-micrometre range

3.9
internal (inner) surface
surface of pores, cavities, or any other heterogeneity within particles or bulk materials

3.10
closed pore
pore totally enclosed by its walls and hence not interconnecting with other pores and not accessible to fluids

[SOURCE: ISO 15901-1:2016, 3.10]

3.11
open pore
pore not totally enclosed by its walls and open to the surface either directly or by interconnecting with other pores and therefore accessible to fluid

[SOURCE: ISO 15901-1:2016, 3.11]

3.12
powder
porous or nonporous solid composed of discrete particles with maximum dimension less than about 1 mm, powders with a particle size below about 1 µm are often referred to as fine powders

[SOURCE: ISO 15901-1:2016, 3.4]

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3.13
granules
granules (granular material) is a conglomeration of discrete solid, macroscopic particles

3.14
monolith
monolith is a single discrete, solid object

4 Symbols

Table 1 — Symbols

Symbol	Description	Unit
S	Total surface area	m^2
S_v	Volume specific surface area (surface to volume ratio)	$m^2\ cm^{-3}$
S_m	Mass specific surface area (surface to mass ratio)	$m^2\ g^{-1}$
m_s	Mass of the scattering sample	g
V	Volume of the scattering sample	cm^3
ρ_s	Density of the sample	$g\ cm^{-3}$
ρ_m	Density of the matrix	$g\ cm^{-3}$
ρ_p	Density of the pore phase or particle	$g\ cm^{-3}$
ρ_{bulk}	Bulk density	$g\ cm^{-3}$
ρ_{grain}	Grain density	$g\ cm^{-3}$
$\rho_{packed\text{-}bed}$	Density of packed beds of nanoparticles	$g\ cm^{-3}$
$\rho_{dispersion}$	Density of dispersion of nanoparticles	$g\ cm^{-3}$
ρ_1	Density of phase 1	$g\ cm^{-3}$
ρ_2	Density of phase 2	$g\ cm^{-3}$
ρ_e	Electron density	nm^{-3}
β	Mass concentration	$g\ cm^{-3}$
q	Momentum transfer, $(4\pi/\lambda)\sin\theta$, with scattering angle 2θ	nm^{-1}
φ_1	Volume fraction of phase 1	
φ_2	Volume fraction of phase 2	
φ_m	Volume fraction of the matrix	
φ_p	Volume fraction of the pore phase (or particle)	
V_p	Mass specific pore volume	cm^3g^{-1}
λ	Wavelength of the incident X-rays	nm
Ω	Solid angle	sr
$d\Sigma/d\Omega$	Macroscopic differential scattering cross-section	$m^{-1}sr^{-1}$
$I(q)$, $I(q)_s$	Scattered intensity of the sample	
$I(q)_{ref}$	Scattered intensity of the reference (standard)	
$\tilde{I}(q)$	Scattered intensity (line-smeared data)	
Q	Invariant	nm^{-3}
$\tilde{Q}$	Invariant (line-smeared data)	
K	Porod constant	
$\tilde{K}$	Porod constant (line-smeared data)	
K_{abs}	Absolute Porod constant	m^{-5}
A	Constant background term	
$\tilde{A}$	Constant background term (line-smeared data)	

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