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Determination of particle concentration by small-angle X-ray scattering (SAXS)

Détermination de la concentration de particules par diffusion des rayons X aux petits angles (SAXS)

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Contents

Page

Foreword ..... **iv**
Introduction ..... **v**
1 Scope **1**
2 Normative references **1**
3 Terms and definitions **1**
4 Symbols and abbreviated terms **2**
5 Principle of the method **3**
5.1 Particle size detection limits 4
5.2 Particle concentration detection limits 4
5.3 Effects of polydispersity 5
6 Apparatus **6**
7 Preliminary procedures and instrument set-up **7**
8 Sample preparation **7**
9 Measurement and data correction procedures **8**
10 Determination of the particle concentration **8**
11 Repeatability **11**
12 Documentation and test report **11**
12.1 Test report 11
12.2 Technical records 12
Annex A (informative) **Inter-laboratory comparison** **13**
Bibliography **14**

Foreword

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

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Small-angle X-ray scattering (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 nanoparticulate systems. Under certain conditions, the upper limit can be extended to 200 nm and beyond. For sufficiently monodisperse spherical particles, the observed oscillations of the scattered intensity as a function of the momentum transfer, which is directly related to the scattering angle and the wavelength of the incident X-rays, enable the size determination of nanoparticles. In order to determine their concentration in a liquid (also called suspending medium, solvent or matrix), the absolute differential scattering cross section has to be determined, thus the ratio of the scattered intensity to the incident intensity. Assumptions on the particle shape are required, which can be based on microscopic techniques like electron microscopy. Furthermore, the electron density difference between the particles and the liquid needs to be known.

The concentration of nanoparticles, thus particles in the size range between about 1 nm to 100 nm, is one of the most important parameters for nanoparticle use in industry, medicine and research, and is expected to become relevant as well for regulatory purposes, especially in the pharmaceutic sector. The application of SAXS for the determination of the mean particle size and size distribution has been described in ISO 17867. This document covers the extension to obtain the nanoparticle concentration as well from SAXS measurements. User-friendly commercial SAXS instruments are available worldwide from several 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 a document that allows users to obtain good inter-laboratory agreement on the accuracy and reproducibility of the technique.

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.

Determination of particle concentration by small-angle X-ray scattering (SAXS)

1 Scope

This document deals with the application of small-angle X-ray scattering (SAXS) for the measurement of the particle concentration in suspensions. In this document, only the concentration of sufficiently monodisperse spherical particles is treated, which means that the width of the size distribution is typically below about 50 % of the mean diameter. Here, the differential scattering cross section can be calculated based on the form factor, which depends only on the momentum transfer q and the particle radius r. Furthermore, this document is limited to 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 needs to be taken and a reduction of the concentration can be necessary.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 17867, *Particle size analysis — Small angle X-ray scattering (SAXS)*

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
particle
minute piece of matter with defined physical boundaries

Note 1 to entry: A physical boundary can also be described as an interface.

Note 2 to entry: A particle can move as a unit.

Note 3 to entry: This definition applies to nano-objects.

[SOURCE: ISO/TS 80004-6:2021, 3.9]

3.2
particle size
linear dimension of a *particle* (3.1) determined by a specified measurement method and under specified measurement conditions

Note 1 to entry: Different methods of analysis are based on the measurement of different physical properties. Independent of the particle property actually measured, the particle size can be reported as a linear dimension, e.g. as an equivalent spherical diameter.

[SOURCE: ISO/TS 80004-6:2021, 4.1.1]

3.3
particle size distribution
distribution of *particles* (3.1) as a function of *particle size* (3.2)

Note 1 to entry: Particle size distribution may be expressed as cumulative distribution or a distribution density (distribution of the fraction of material in a size class, divided by the width of that class).

[SOURCE: ISO/TS 80004-6:2021, 4.1.2]

3.4
suspension
heterogeneous mixture of materials comprising a liquid and a finely dispersed solid material

[SOURCE: ISO/TS 80004-6:2021, 3.13]

3.5
concentration
amount-of-substance of a component divided by the volume of the system

[SOURCE: ISO 18113-1:2022, 3.2.12]

3.6
particle number concentration
number of particles per unit of volume of suspension

Note 1 to entry: The particle number concentration can also be given as number of particles per unit of mass of suspension. Literature values for the density of the liquid can be used for the conversion as, in most cases, the low content of particles for which this document is applicable will not affect the sample density significantly.

[SOURCE: ISO 29464:2017, 3.2.131]

4 Symbols and abbreviated terms

The symbols and abbreviated terms used in this document are listed in Table 1.

Table 1 — Symbols

Symbol	Description	Unit (with prefix)
C	Particle number concentration	l^{-1}
$\bar{d}_{\ln}$	Median of lognormal size distribution	nm
$\bar{d}_{num}$	Number-weighted mean particle diameter	nm
f_1, f_2	Atomic scattering factors	
$g_{num}(r)$	Number-weighted particle size distribution	
I_{in}	Primary beam intensity without sample	
$I(q)$	Scattered intensity (or scattering intensity)	
M	Molar mass	g/mol
N	Number of particles	
N_A	Avogadro constant	mol^{-1}
$P(q, r)$	Particle form factor as functions of q-value and particle radius, r	
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_e	Thomson radius	fm
$S(q,r)$	Structure factor as functions of q-value and particle radius, r	
T	Transmission	

Table 1 *(continued)*

Symbol	Description	Unit (with prefix)
t_o	Optimum sample thickness	mm
w	Sample thickness	mm
Z	Number of protons	
λ	Wavelength of the incident X-rays in vacuum	nm
μ	Linear absorption coefficient	mm^{-1}
ρ	Mass density	g/cm^3
ρ_e	Electron density	nm^{-3}
ρ_{e_P}	Electron density of particles	nm^{-3}
ρ_{e_L}	Electron density of the liquid	nm^{-3}
$\Delta\rho_e$	Electron density difference	nm^{-3}
σ	Standard deviation of Gaussian size distribution	nm
$\frac{d\Sigma}{d\Omega}(q)$	Differential scattering cross section per volume	cm^{-1} sr^{--1}
σ_{ln}	Standard deviation of logarithm of particle size distribution	
2θ	Scattering angle	deg or rad
Ω	Solid angle of a detector pixel	sr

5 Principle of the method

When X-rays pass through matter, a small fraction of the radiation can be scattered due to electron density differences in the matter. The scattered radiation intensity profile (as a function of the scattering angle or momentum transfer, q), contains information that can be used to deduce morphological characteristics of the material. In the small-angle regime (typically $2\theta < 5°$; wavelength dependent), information on the particle dimensions within the material is available from the elastic scattering arising from the electron density contrast between the particles and the medium in which they reside, typically a liquid. For sufficiently monodisperse spherical nanoparticles, the scattering pattern consist of concentric rings, corresponding to oscillations of the scattered intensity as function of the scattering angle or momentum transfer, q. If the (electron) density of the particles and the surrounding liquid are known, the nanoparticle concentration can be determined by comparing the calculated and the measured differential scattering cross section. The method requires the calibration of the q-axis and the intensity axis. The absolute scattering cross section can be obtained by using either:

— primary or secondary standards such as water, Lupolen or glassy carbon with calculable or known scattering cross section;

— an area detector with very high dynamic range (such as hybrid-pixel detectors) so that the incident radiation (direct beam) and the scattered radiation can be measured;

— an area detector with known quantum efficiency for the scattered radiation and an additional detector (such as a calibrated photodiode) to determine the incident photon flux.

Calibration with reference materials consisting of nanoparticles with known concentration is not required in these cases.

If an absolute intensity calibration is not possible, it is still possible to determine the mass concentration from the extrapolated forward scattering intensity using a similar monodisperse reference material with known mass concentration.

At increased concentrations, i.e. those higher than ten 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 this document. In practice, a concentration ladder may be explored 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 allow identification of concentration artefacts.

5.1 Particle size detection limits

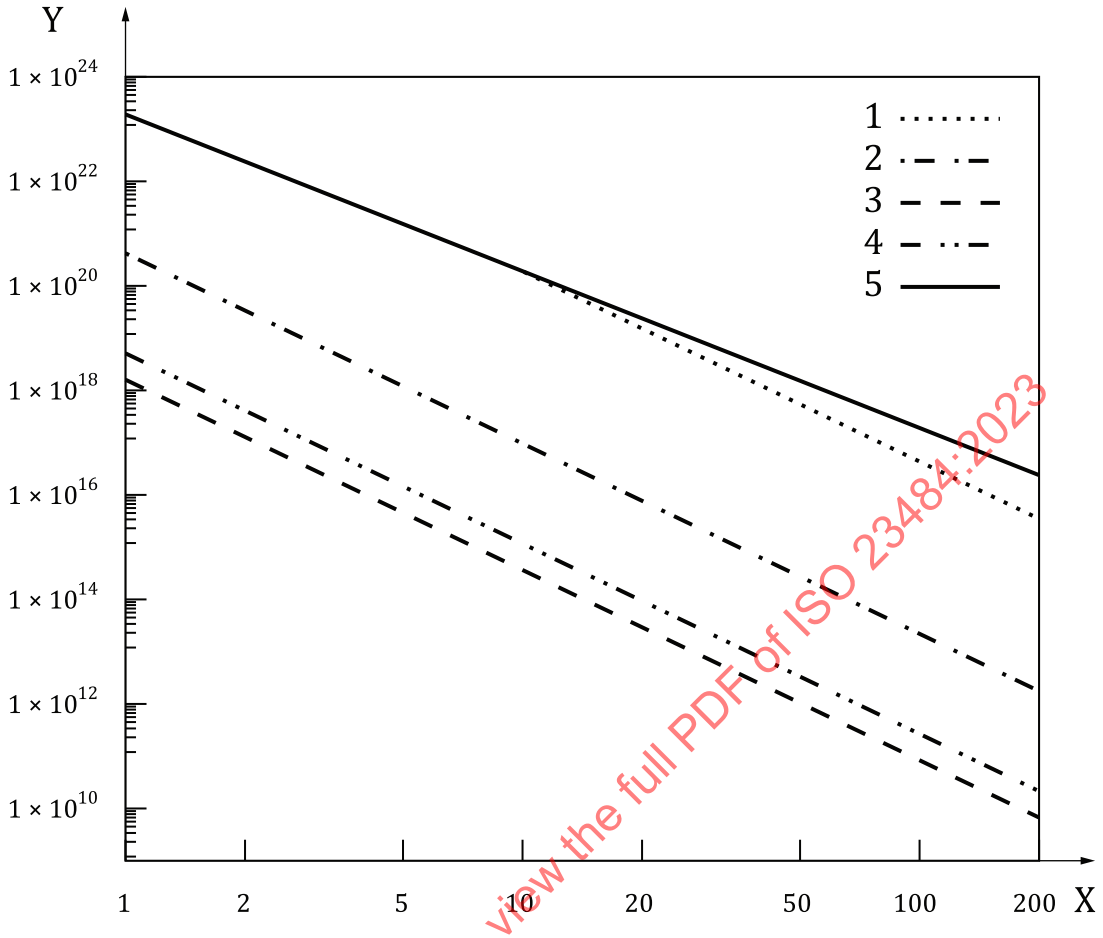
The determination of the particle size (mean particle diameter) and the size distribution shall be in accordance with ISO 17867. The accessible size range strongly depends on the instrument. In order to register at least one minimum in the scattered intensity as function of the momentum transfer, the lower diameter limit is typically a few nm. The higher diameter limit is about hundred nm for most laboratory instruments, but the range can be extended to several hundred nm at dedicated ultra small angle X-ray scattering (USAXS) instruments which are available at some synchrotron radiation facilities and some laboratory instruments.

5.2 Particle concentration detection limits

The particle number concentration limits vary as well with the instrument, but even more with the size and the (electron) density of the particles. The scattered intensity of spherical particles scales with the sixth power of the particle size, thus the accessible lower number concentration limit of large particles is orders of magnitude lower. On the other hand, the scattered intensity scales with the square of the electron density difference between particles and liquid. Therefore much lower concentration ranges are accessible for gold nanoparticles compared to polystyrene particles. Table 2 provides typical orders of magnitude for the lower limit of detection (LLD) for nanoparticles of different diameters and materials suspended in water. The accessible concentration ranges are schematically shown in Figure 1.

Table 2 — Lower limit of detection for nanoparticle number concentration

Material	Density g/cm^3	Diameter nm	LLD number concentration l^{-1}
Polystyrene	1,05	10	10^{20}
Polystyrene	1,05	100	10^{17}
Silica	2,65	10	10^{17}
Silica	2,65	100	10^{14}
Silver	10,49	10	10^{15}
Silver	10,49	100	10^{12}
Gold	19,28	10	10^{14}
Gold	19,28	100	10^{11}



Key

X diameter/nm

Y number of particles/litre

1 lower limit for polystyrene particles in water

2 lower limit for silica particles in water

3 lower limit for gold particles in water

4 lower limit for silver particles in water

5 upper limit for all particles corresponding to 10 % volume fraction

NOTE For low-density particles like polystyrene, the low concentration limit can be decreased, for example, by using ethanol instead of water as liquid.

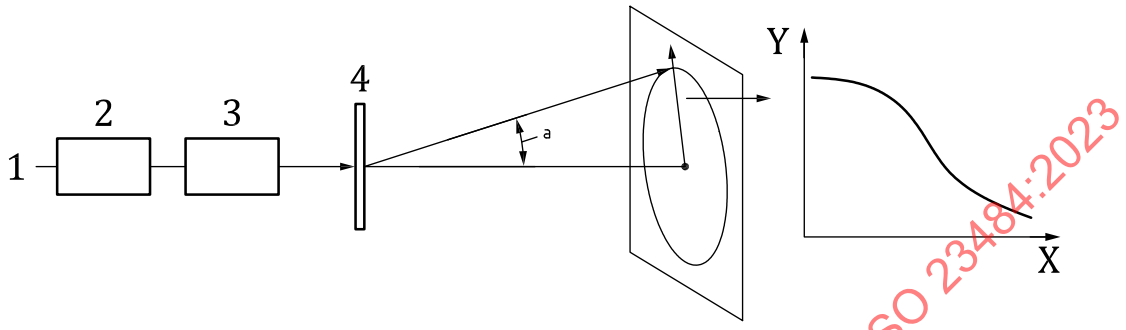
Figure 1 — Schematic representation of the accessible concentration ranges for spherical nanoparticles of four different materials (gold, silver, silica and polystyrene) in aqueous suspension as function of the particle diameter

5.3 Effects of polydispersity

The issue of polydispersity is extremely important for most real samples. As mentioned above, the scattered intensity is proportional to the sixth power of the radius, thus the scattering from larger particles can hamper the detection of smaller size fractions in polydispersed samples. Thus, the samples have to be sufficiently monodisperse. If the size distribution is too broad, minima are no longer observed in the scattering curve, and thus a unique model fitting is no longer possible. Also, a size distribution asymmetry, thus a non-Gaussian or non-lognormal size distribution would lead to deviations for the particle concentration.

6 Apparatus

The general design of a SAXS instrument is shown in Figure 2. The SAXS set-up consists of X-ray source, optics, collimation system, sample holder, beam stop and detector. The greatest challenges in SAXS are to separate the parasitic scattering from the collimation system and the unscattered, transmitted beam ("direct beam") from the scattered radiation at small angles (around 0,1°). The direct beam is normally blocked by a beam stop and parasitic scattering should be eliminated. The need for separation of primary and scattered beam makes collimation of the primary beam mandatory.

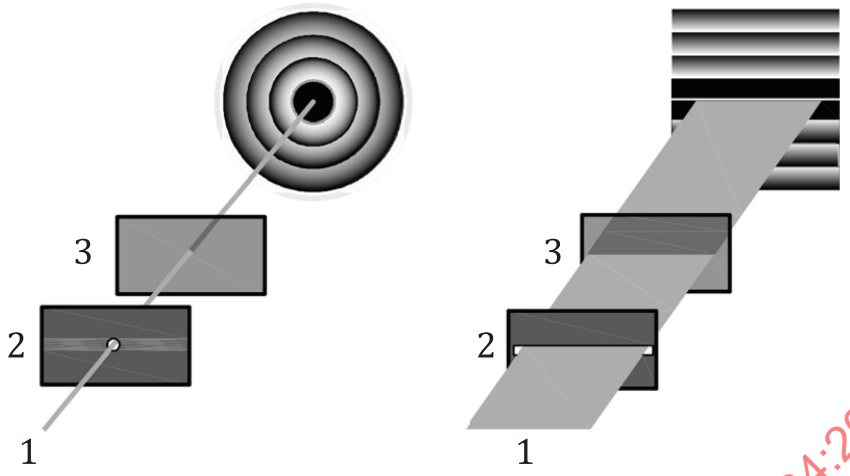


Key

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

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

The above outlined principles strictly apply only for scattering patterns that are obtained with ideal point-collimation optics, i.e. point shaped X-ray beam cross section, and monochromatic radiation. However, a widely popular camera design (Kratky camera or slit collimators) uses line collimation, i.e. the probing X-ray beam is a narrow ribbon with line-shaped cross section. This has the advantage of producing higher intensities in the weak outer part of the scattering curve (towards large q), but generally requires numerical corrections (desmearing). The two design principles are shown in Figure 3.



Key

1 X-ray source
2 collimation system
3 sample

Figure 3 — Schematic view of point- (left) and line-collimation (right) optics

7 Preliminary procedures and instrument set-up

The momentum transfer q is related to the scattering angle and the wavelength. Wavelength calibration can be performed before conducting an experiment and thus would be classified as a preliminary procedure, but this is only required for polychromatic sources. If characteristic X-ray emission lines (e.g. copper Kα or molybdenum Kα lines) are used, a suitable absorber can be used to check that the right emission line has been selected correctly (nickel for Cu Kα, zirconium for Mo Kα). The scattering angle follows from the geometry of the experimental set-up and can be obtained from the pixel size of the X-ray detector and the sample-to-detector distance. If the latter can be varied, the distance can be determined by triangulation.

The utilization of a calibration material for the q-value, such as silver behenate, is a simple and convenient alternative to calibrate the q-axis.

For the determination of the particle number concentration, the scattered intensity shall be scaled to absolute units. For this purpose, a variety of auxiliary calibration materials are available, including water and glassy carbon. Alternatively, many current SAXS instruments can determine this directly by means of calibrated detectors, or measurement of the unattenuated primary beam intensity if an area detector with very high linearity is used. Otherwise, or if the incident photon flux is very high (e.g. at synchrotron radiation beamlines), it is also possible to determine the incident photon flux (e.g. with a calibrated ion chamber or photodiode) and to use the previously determined quantum efficiency of the area detector. The use of semi-transparent beamstops to measure the beam intensity is not recommended due to the radiation hardening effects of such, which can lead to inaccurate values.

All calibrations should be described in the analysis report.

8 Sample preparation

Sample preparation is simple and fast for SAXS measurements. The required sample volumes are small, typically in a range of 5 μl to 50 μl for liquids, if copper radiation is used.

Liquid samples are usually measured inside a thin-walled sample cell, typically a capillary with a diameter between 0,5 mm to 2 mm when the liquid primarily contains water or hydrocarbons. Particles in a liquid that contains heavy atoms, for example, chlorine in chloroform, should be measured

in smaller diameter capillaries as the atoms strongly absorb the incident radiation, or higher energy radiation should be used. The sample thickness has to be determined, which is easier for capillaries with rectangular cross-section, but their wall thickness is usually larger.

It is strongly recommended to measure liquid samples in a re-fillable, well characterized or flow-through sample cell, as this greatly reduces the risks of errors encountered in the background subtraction procedure (incorrigible effects may lead to inaccurate subtraction if non-identical sample cells are used for the two measurements).

9 Measurement and data correction procedures

If possible, a SAXS particle-concentration experiment should consist of at least two measurements using the same sample holder and preferably the same acquisition time:

a) a background measurement (containing signals from the liquid or matrix, the (same) sample cell windows, the parasitic instrument radiation, background radiation and detector noise);

b) a sample measurement (containing signals from the particles, the liquid or matrix, the sample cell windows, the parasitic instrument radiation, background radiation and detector noise).

Care shall be taken that the scattering of the window material of the sample cell, the parasitic scattering of the SAXS instrument and the dark count rate of the detector are removed. The transmission from the sample and background/liquid and efficiency variation over the detector shall be taken into account.

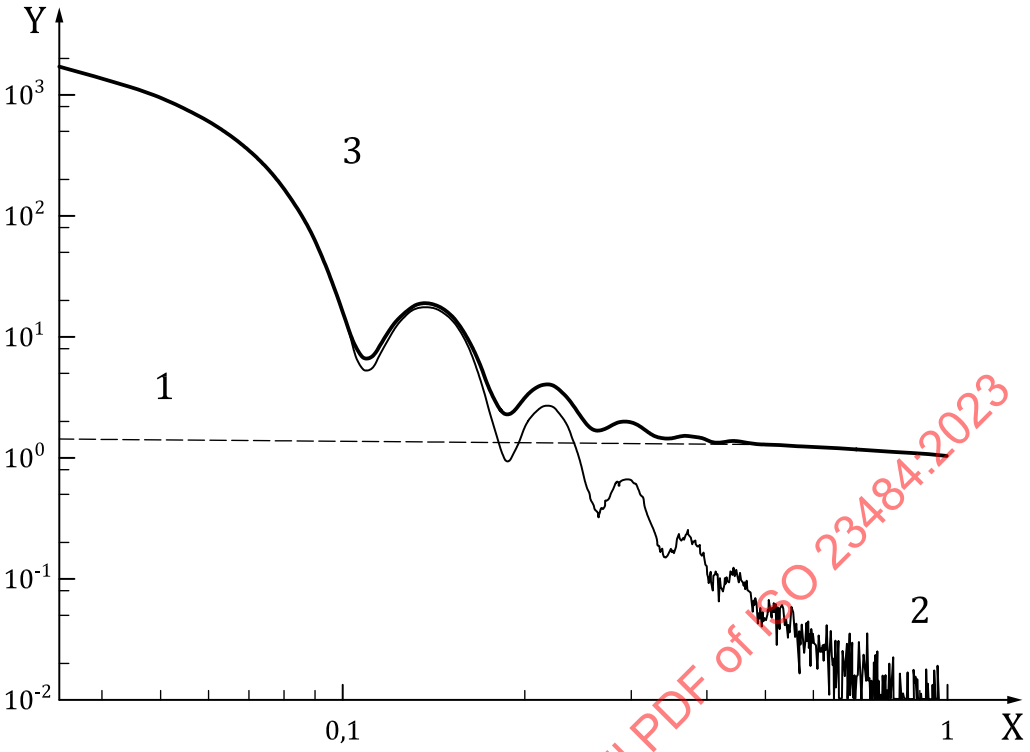
The statistical quality of the scattering pattern improves with increasing intensity and complies with standard statistics for signals obtained by the subtraction of two independent measurements.

As the data analysis methods are sensitive to the quality of the data, the scattering signal of the sample shall be carefully extracted from the total measured signal. These correction steps are performed by the software provided with commercial instruments or through custom software. At best, they propagate the data uncertainties through the correction steps, resulting in a final data set complete with uncertainty estimates on the data points.

The minimum amount of data corrections to be considered or applied by this software to the data before background subtraction are corrections for: invalid pixels, dark counts, time, X-ray flux and X-ray transmission. This is followed by the background subtraction (complying with standard statistics for signals obtained by the subtraction of two independent measurements). After the background subtraction, a normalization for sample thickness can be performed, as well as the scaling to the absolute differential scattering cross section per volume.

10 Determination of the particle concentration

The integration of the pattern registered with the X-ray detector leads to the scattered intensity as a function of the momentum transfer or q-value, which is the magnitude of the scattering vector and is given by $q = (4\pi/\lambda) \sin\theta$, where λ is the wavelength of the incident monochromatic X-rays and θ is half of the scattering angle. A typical example showing the sample scattering, the scattering of the liquid or matrix and the difference (the corrected signal only due to particle scattering) is given in Figure 4.



Key

X q (in nm^{-1})
Y scattered intensity (in arbitrary units)
1 scattering from liquid
2 scattering from particles
3 scattering from suspension

Figure 4 — Typical SAXS profiles of a suspension, the liquid and the difference (the corrected signal only due to particle scattering)

The differential scattering cross section per volume $\frac{d\Sigma}{d\Omega}(q)$ as a function of the momentum transfer q can be expressed according to Formula (1) as the sum of the scattering from an ensemble of particles:

$$\frac{d\Sigma}{d\Omega}(q) = r_e^2 \cdot C \cdot \Delta\rho_e^2 \int_0^\infty g_{num}(r) S(q,r) |P(q,r)|^2 dr, \quad (1)$$

where r_e is the Thomson radius, C the particle number concentration, thus the number of scatterers (e.g. nanoparticles) per volume, $g_{num}(r)$ is the size distribution function, $S(q,r)$ is the structure factor, $P(q,r)$ is a form factor and $\Delta\rho_e = \rho_{e\_P} - \rho_{e\_L}$ is the electron density difference or contrast of the SAXS experiment.

Here, ρ_{e_P} is the electron density of the particles and ρ_{e_L} is the electron density of the liquid or matrix. The electron density of a substance can be calculated according to Formula (2) by:

$$\rho_e = (\rho \cdot Z \cdot N_A) / M \quad (2)$$

where

Z is the number of protons;

N_A is the Avogadro constant;

M is the molar mass.

For water at room temperature, use $\rho_{e\_L} = (333{,}5 \pm 0{,}3)\ \mathrm{nm}^{-3}$.

The accuracy can be increased by using the effective electron density which takes the slight photon energy dependence of the contrast into account by introducing the energy-dependent atomic scattering factors. Z is then replaced by $(f_1(E_{Ph}) + i^* f_2(E_{Ph}))$.

For nanoparticle suspensions with particle concentrations below about 1 vol %, the structure factor can be neglected, thus $S(q,r) = 1$. If the nanoparticles are sufficiently monodisperse and spherical, the corresponding form factor can be used according to Formula (3):

$$P(q,r) = \frac{4}{3}\pi r^3 \left(3\frac{\sin(qr) - qr\cos(qr)}{(qr)^3} \right). \tag{3}$$

The most common distributions are Gaussian and lognormal. A Gaussian distribution is given in Formula (4) by:

$$g_{num}(r) = \exp\left(-\frac{(r - \bar{d}_{num}/2)^2}{\sigma^2/2} \right) \Big/ \int_0^\infty \exp\left(-\frac{(r - \bar{d}_{num}/2)^2}{\sigma^2/2} \right) dr \tag{4}$$

and a lognormal distribution can be written as in Formula (5):

$$g_{num}(r) = \frac{1}{\sqrt{2\pi} r \sigma_{ln}} \exp\left(-\left(ln\left(\frac{2r}{\bar{d}_{ln}} \right) \right)^2 / 2\sigma_{ln}^2 \right) \tag{5}$$

where the mean diameter $\bar{d}_{ln}$ can be transformed to the number-weighted mean particle diameter $\bar{d}_{num}$ according to Formula (6):

$$\bar{d}_{num} = \bar{d}_{ln} exp\left(\sigma_{ln}^2/2 \right) \tag{6}$$

where σ (or σ_{ln}) is the standard deviation of the size distribution.

Using the measured scattered intensity $I(q)$ and other experimentally accessible parameters like the transmission of the sample T, the almost constant solid angle Ω of a detector pixel and the sample thickness w, the differential scattering cross section per volume can be calculated according to Formula (7) as:

$$\frac{d\Sigma}{d\Omega}(q) = \frac{I(q)}{I_{in} \cdot T \cdot \Omega \cdot w} \tag{7}$$

The combination of the equations leads to Formula (8):

$$\frac{I(q)}{I_{in} \cdot T \cdot \Omega \cdot w} = r_e^2 \cdot C \cdot \Delta\rho_e^2 \int_0^\infty g_{num}(r) \cdot |P(q,r)|^2 dr. \tag{8}$$

The mean particle diameter $\bar{d}_{num}$, the standard deviation σ - both part of $g_{num}(r)$ - and the particle number concentration C are obtained from a fit of this formula to the measured data.

It is also possible to use Monte Carlo- or expectation maximization- based data fitting as for the size determination. If a method only provides a volume-weighted concentration, the conversion to the particle number concentration is not recommended.

For sufficiently monodisperse, spherical particles for which the electron density is known and well above the electron density of the liquid (e.g. for gold or silver particles in water), relative uncertainties for the number concentration below 10 % can be achieved. For gold nanoparticles in water, agreements within 3 % have been achieved (see Annex A). As the obtained result depends on the square of the electron density difference, the uncertainty can be significantly higher for particles with lower density like silica or polystyrene.

11 Repeatability

Repeated measurements of the same sample can indicate if the material is changing during the duration of the experiment and therefore can be an indicator of degradation under the X-ray beam. A major problem in concentration measurement can be sedimentation. If static capillaries are used, measurements at different positions along the capillary axis can be performed to study this effect. Additionally, sample-to-sample measurements will indicate homogeneity or heterogeneity of the material. Sample-to-sample heterogeneity and instability of a sample/material over time can only be detected if the heterogeneity and instability create effects that can be distinguished beyond the method repeatability.

12 Documentation and test report

12.1 Test report

Test reports should contain the following information.

a) A reference to this document, i.e. ISO 23484:2023.

b) The mean particle diameter $\bar{d}_{\mathrm{num}}$ and its uncertainty, including a clear statement whether this represents a number, volume or intensity weighted mean. In the absence of a full uncertainty evaluation, the standard deviation from several repeated measurements should be provided as estimate of the repeatability. ISO/IEC Guide 98-3 can assist here, but expert judgment should be employed.

c) The determined particle number concentration and, if available, its uncertainty.

d) Complete sample identification, including available information on particle shape and homogeneity. Electron micrographs, where relevant and informative, can be included in order to convey information on particle shape, degree of dispersion, crystallinity and other visual indicators that are not easily conveyed in graphical or tabular data.

e) Applied data evaluation, including a reference to the clause which explains how the results were calculated.

f) Form factor and size distribution.

g) Used electron density of the particles and the liquid.

h) Range of q selected for evaluation.

i) Any deviations from the procedure.

j) Any unusual features observed.

k) The date of the test.