INTERNATIONAL STANDARD

ISO 13696

Second edition
2022-06

Optics and photonics — Test method for total scattering by optical components

Optique et photonique — Méthodes d'essai du rayonnement diffusé par les composants optiques

Reference number
ISO 13696:2022(E)

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Published in Switzerland

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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 of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 172, *Optics and Photonics*, Subcommittee SC 9, *Laser and electro-optical systems,* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 123, *Lasers and photonics*, in accordance with the agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 13696:2002), which has been technically revised.

The main changes are as follows:

— In the Scope, measurement range outlined in more detail and limited to 250 nm. For measurements in the deep ultraviolet between 190 nm to 250 nm, specific methods are considered and are described.

— In 3.1.6, additional Note 2 inserted for high volume scattering of the specimen and additional Note 3 inserted for comprehensive illustration of the term total scattering.

— In 3.1.7, Note extended concerning diffuse reflectance standard for wavelengths below 250 nm down to the deep ultraviolet.

— In 3.2, New symbols for total scattering, σ_{TS}, forward scattering, τ_{TS}, and backward scattering, ρ_{TS}, in Table 1.

— In Figure 1 and 4.2.5, lock-in amplifier optional. For fast data acquisition modules, no Lock-in technique may be necessary.

— In 4.2.2, calibration of the monitor detector is not necessary. The power at the sample surface shall be measured by a calibrated detector.

— In 4.2.4, additional Note 1 inserted concerning aging of the diffuse reflecting material on the inner walls of the sphere.

— In 4.2.5, additional Note inserted concerning optional components for a phase sensitive detection scheme with lock-in amplifier.

- In 5.3, change of measurement sequence starting with power measurement calibration procedure, and determination of the signal of the unloaded sphere prior to the measurement of the specimen.
- In 6.1, adaptation of Formulae (1) (2) and (5) to (8) (in the denominator $V_c(r_i)$ was adapted to V_c).
- Correction of Formula (C.2).
- Annex E inserted concerning alternative method for calibrating total scatter measurements using a calcium fluoride diffuser disk.
- In Bibliography, ISO 31-6:1992 was replaced by current version ISO 80000-7, same for ISO 11146 with ISO 11146-1 and ISO 11146–2, ISO 11554 and ISO 12005 no longer cited dated. Also replacement of former citations "[5]" by latest edition of SEMI MF1048-0217[6].

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

In most applications, scattering in optical components reduces the efficiency and deteriorates the image-forming quality of optical systems. Scattering is predominantly produced by imperfections of the coatings and the optical surfaces of the components. Common surface features, which contribute to optical scattering, are imperfections of substrates, thin films and interfaces, surface and interface roughness, or contamination and scratches. These imperfections deflect a fraction of the incident radiation from the specular path. The spatial distribution of this scattered radiation is dependent on the wavelength of the incident radiation and on the individual optical properties of the component. For most applications in laser technology and optics, the amount of total loss produced by scattering is a useful quality criterion of an optical component.

This document describes a testing procedure for the corresponding quantity, the total scattering value, which is defined by the measured values of backward scattering or forward scattering. The measurement principle described in this document is based on an Ulbricht sphere as the integrating element for scattered radiation. An alternative apparatus with a Coblentz hemisphere, which is also frequently used for collecting scattered light, is described in Annex A.

Optics and photonics — Test method for total scattering by optical components

1 Scope

This document specifies procedures for the determination of the total scattering by coated and uncoated optical surfaces. Procedures are given for measuring the contributions of the forward scattering or backward scattering to the total scattering of an optical component.

This document applies to coated and uncoated optical components with optical surfaces that have a radius of curvature of more than 10 m. Measurement wavelengths covered by this document range from the ultraviolet above 250 nm to the infrared spectral region below 15 µm. For measurements in the deep ultraviolet between 190 nm to 250 nm, specific methods are considered and are described. Generally, optical scattering is considered as neglectable for wavelengths above 15 µm.

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 11145, *Optics and photonics — Lasers and laser-related equipment — Vocabulary and symbols*

ISO 14644-1, *Cleanrooms and associated controlled environments — Part 1: Classification of air cleanliness by particle concentration*

3 Terms, definitions and symbols

3.1 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 11145 and the following 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/

3.1.1
scattered radiation
fraction of the incident radiation that is deflected from the specular optical path

3.1.2
front surface
optical surface that interacts first with the incident radiation

3.1.3
rear surface
surface that interacts last with the transmitted radiation

3.1.4
backward scattering
fraction of radiation scattered by the optical component into the backward halfspace

Note 1 to entry: Backward halfspace is defined by the halfspace that contains the incident beam impinging upon the component and that is limited by a plane containing the front surface of the optical component.

3.1.5
forward scattering
fraction of radiation scattered by the optical component into the forward halfspace

Note 1 to entry: Forward halfspace is defined by the halfspace that contains the beam transmitted by the component and that is limited by a plane containing the rear surface of the optical component.

3.1.6
total scattering
ratio of the total power generated by all contributions of *scattered radiation* (3.1.1) into the forward or the backward halfspace to the power of the incident radiation

Note 1 to entry: The halfspace in which the scattering is measured should be clearly stated.

Note 2 to entry: The sum of the measured forward and backward scattering does not include the contribution of the bulk material in the optical component. In case the volume scattering of the component is not negligible, the total scatter losses may exceed the sum of forward and backward scattering.

Note 3 to entry: Total scattering is equal to forward or backward scattering, and is neither the sum of both nor the sum of all scattering contributions.

3.1.7
diffuse reflectance standard
diffuse reflector with known total reflectance

Note 1 to entry: Commonly used diffuse reflectance standards are fabricated from barium sulfate or polytetrafluoroethylene powders (see Table 2). The total reflectance of reflectors freshly prepared from these materials is typically greater than 0,98 in the spectral range given in Table 2, and it can be considered as a 100 % reflectance standard. For increasing the accuracy, diffuse reflectance standards with lower reflectance values can be realized by mixtures of polytetrafluoroethylene powder and powders of absorbing materials, see Reference [6]. Further concepts for diffuse reflectance standards include optical surfaces with specially prepared microstructures, metal-coated diffusers or diffuse transparent reference samples. A versatile method on the basis of a calcium fluoride diffuser disk for the wavelength range from 250 nm down in the ultraviolet range is described in Annex E.

3.1.8
range of acceptance angle
range of scattering angles in the reflecting or transmitting hemisphere, which are collected by the integrating element

Note 1 to entry: The maximum polar acceptance angle with respect to the sample normal is 85°.

Note 2 to entry: The radiant power around the specular transmitted or reflected beam is not collected by the integrating element in a cone with an opening angle of 2° or less.

3.1.9
angle of polarization
angle between the major axis of the instantaneous polarization ellipse of the incident radiation and the plane of incidence

Note 1 to entry: For non-normal incidence, the plane of incidence is defined by the plane which contains the direction of propagation of the incident radiation and the normal at the point of incidence.

Note 2 to entry: The angle of polarization, γ, is identical to the azimuth, Φ (according to ISO 12005), if the reference axis is located in the plane of incidence.

3.2 Symbols and units of measure

Table 1 — Symbols and units of measure

Symbol	Term	Unit
λ	wavelength	nm
α	angle of incidence	degrees
γ	angle of polarization	degrees
d_σ	beam diameter on the surface of the specimen	mm
$d_{\sigma,\mathrm{p}}$	largest beam diameter at a beam port	mm
P_{inc}	power of the incident radiation	W
P_{bac}	total power, backward scattered radiation	W
P_{for}	total power, forward scattered radiation	W
σ_{TS}	total scattering	
ρ_{TS}	backward scattering	
τ_{TS}	forward scattering	
$V_{\mathrm{s,bac}}$	detector signal for the specimen, backward scattering	[a]
$V_{\mathrm{s,for}}$	detector signal for the specimen, forward scattering	[a]
V_{c}	detector signal, diffuse reflectance standard	[a]
V_{u}	detector signal, test ports open	[a]
τ_{s}	transmittance of specimen at wavelength, λ	
ρ_{s}	reflectance of specimen at wavelength, λ	
r_i	test site position	
N	number of test sites per surface	

[a] The unit depends on the measurement device and is therefore not specified here.

4 Test method

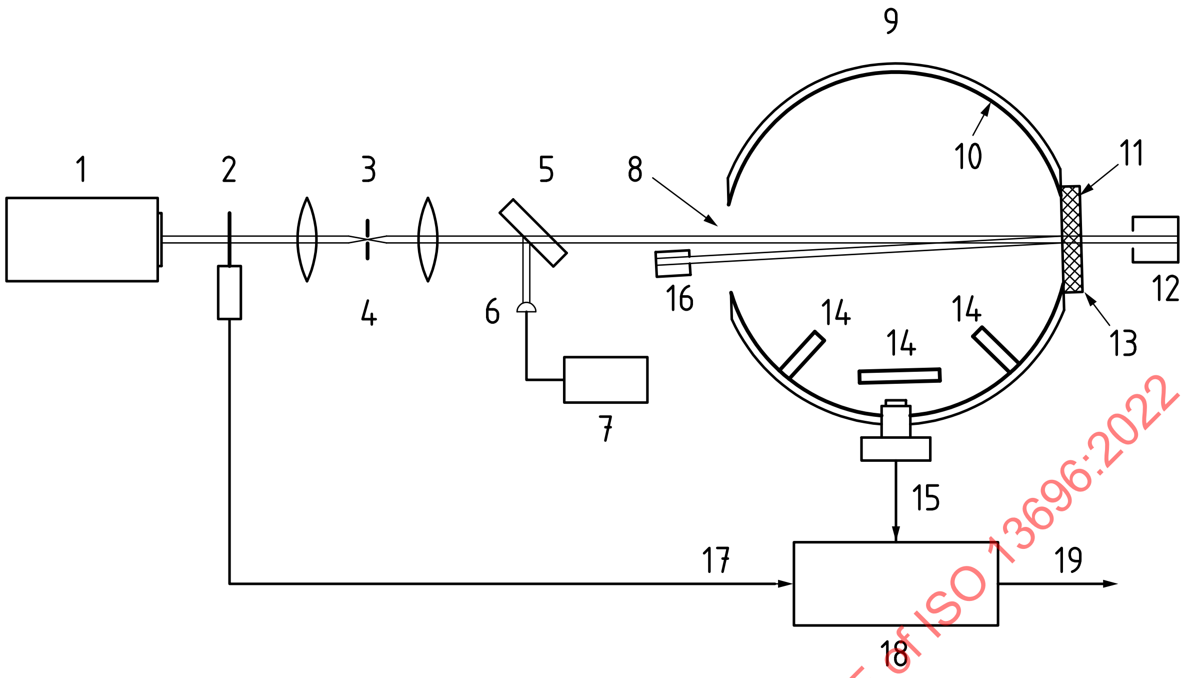
4.1 Principle

The fundamental principle (see Figure 1) of the measurement apparatus is based on the collection and integration of the scattered radiation. For this purpose, a hollow sphere with a diffusely reflecting coating on the inner surface (Ulbricht sphere) is used. Beam ports are necessary for the transmission of the test beam and the specularly reflected beam through the wall of the sphere. The sample is attached to one of these ports forming a part of the inner surface of the sphere. For the measurement of the backward scattering, the specimen is located at the exit port. The forward scattering is determined by mounting the specimen to the entrance port. The scattered radiation is integrated by the sphere and measured by a suitable detector, which is attached to an additional port at an appropriate position. A diffuse reflectance standard is used for calibration of the detector signal.

4.2 Measurement arrangement and test equipment

4.2.1 General

The measurement facility used for the determination of the total scattering is divided into four functional sections, which are described in detail below. One functional section consists of the radiation source and the beam preparation system. Two different components are defined by the integration and detection of the scattered radiation. Another section is formed by the sample holder and its optional accessories.



Key

1 radiation source
2 chopper
3 spatial filter
4 beam splitter
5 power detector
6 power meter
7 entrance port
8 Ulbricht sphere
9 coating
10 exit port
11 beam stop
12 sample
13 radiation baffles
14 detector, diffuser
15 beam stop
16 chopper signal
17 lock-in amplifier (optional)
18 detector signal

Figure 1 — Schematic arrangement for the measurement of total scattering
(configuration for backward scattering with phase sensitive detection scheme)

4.2.2 Radiation source

As radiation sources, lasers are preferred because of their excellent beam quality and the high power density achievable on the sample surface. For special applications, for example involving the wavelength dependence of scattering, different conventional radiation sources may be used.

The temporal power variation of the radiation source shall be measured and documented. For this purpose, a beam splitter and a monitor detector are installed. The power at the sample surface shall be measured by a calibrated detector for both test locations at the entrance and exit port of the integrating element.

4.2.3 Beam preparation system

The beam preparation system consists of a spatial filter and additional apertures, if necessary, for cleaning the beam. For measurements involving conventional radiation sources, additional optical elements are required for the shaping and collimation of the beam. The beam diameter, d_σ, at the surface of the specimen shall be greater than 0,4 mm. No radiation power shall be present in the collimated beam profile beyond radial positions exceeding the beam radius by a factor of 5.

NOTE 1 The behaviour of the measured total scatter value can be dependent on the beam diameter and the beam profile (see Annex D).

On the sample surface, the beam profile shall be smooth without local power density values exceeding the average power density within the beam diameter, d_σ, by a factor of three. For measurement systems with a laser as the radiation source, a TEM_{00}-operation with a diffraction-limited Gaussian beam profile is recommended. The defined state and angle of polarization shall be selected. For measurement systems using conventional radiation sources, an unpolarised beam with a circular profile shall be realized. The beam profile on the sample surface shall be free of diffraction patterns and parasitic spots in the outward region. The spatial beam profile on the sample surface shall be recorded and documented.

Optical elements, as for example beam deflection mirrors or beam splitters, may have a reflectivity which depends on the polarization state of the incident radiation, and they may also deteriorate the sensitivity of the measurement. The last optical element in front of the integrating sphere shall be positioned such that the measurement is not influenced by it.

For the fractions of the beam reflected and transmitted by the sample, efficient beam dumps shall be used to suppress backscattering into the integrating sphere.

NOTE 2 An efficient beam dump can be constructed with a stack of optically absorbing neutral density filters. These filters are arranged for non-normal angles of incidence in a housing with optically absorbing inner walls.

4.2.4 Integrating sphere

An integrating sphere is used for the collection and integration of the radiation scattered by the sample. The sphere shall be equipped with beam ports for the entrance and the exit of the probe beam and the fraction of the beam which is specularly reflected by the specimen. The inner surface shall be coated with a highly diffusive reflecting material with a Lambertian characteristic and diffuse reflectivity higher than 97 % for the measurement wavelength. Selected materials suitable for this coating and the corresponding spectral ranges are listed in Table 2.

NOTE 1 Aging of the diffuse reflecting material on the inner walls of the sphere can occur. Corresponding effects can be detected by monitoring the signal of the sphere with attached diffuse reflectance standard during long term usage.

Table 2 — Selected materials for coating of the inner surface of the integrating sphere and for diffuse reflectance standards

Material	Spectral range µm
Barium sulfate	0,35 to 1,4
Magnesium oxide	0,25 to 8,0
Polytetrafluoroethylene	0,20 to 2,5
Gold coating, matt	0,70 to 20

The diameters of the beam ports shall be equal and shall exceed the largest beam diameter, $d_{\sigma,p}$, of the probe beam at the beam ports by at least a factor of five. The port for the detector shall be adapted to the sensitive area of the detecting element. The detailed shape of the ports shall be optimized for minimum deterioration of the integrating action and for a contact-free installation of the test sample. Baffles coated with the same material as the inner surface of the sphere shall be installed between the detector port and the exit as well as the entrance port. Radiation baffles in front of the detector port are recommended in order to shield the detector against radiation directly scattered by the specimen to the location of the detector. For compensation of spatial inhomogeneities of the detector sensitivity, an optional diffuser may be attached to the detector.

An interval from 2° to 85° is defined as the minimum range of the acceptance angle for scattered radiation. The minimum size of the integrating sphere is specified by the lower limit of 2,0° for the acceptance angle.

NOTE 2 The determination of the minimum size of the integrating sphere originates from the largest beam diameter, $d_{\sigma,p}$, at the beam ports of the Ulbricht sphere. The minimum diameter of the port, where the beam diameter appears with largest value $d_{\sigma,p}$ is directly related to this beam diameter by the factor of five. The minimum sphere diameter is then calculated on the basis of the minimum diameter of the entrance port and the lower limit for the acceptance angle. (The minimum diameter of the integrating sphere is at least 72 times the beam diameter, $d_{\sigma,p}$.)

For measurement systems with radiation sources other than lasers or special measurement conditions, the beam diameter, $d_{\sigma,p}$, achievable may result in an impractically large size of the integrating sphere. In such cases, the diameters of the entrance and exit ports shall be adjusted to a value that guarantees no vignetting of the incident, transmitted and reflected beams. The lower and upper limits for the acceptance angles shall be documented.

For specific problems caused by limitations of the integrating element, the detectors and radiation source shall be taken into account for an application of this document below a wavelength of 250 nm. The amount of radiation scattered is a function of both the different contributions of scattering mechanism acting in the specimen and the wavelength of the radiation. In practice, scattering becomes less important at longer wavelengths.

As an alternative, a Coblentz half-sphere with an appropriate reflecting surface may be used. A typical set-up and the corresponding measurement procedure are described in Annex A.

4.2.5 Detection system

For detection of the scattered radiation, a detector is used that is appropriate for the wavelength range of the radiation source. The detector system shall have a sufficient sensitivity for the radiation source and a dynamic range greater than 10^5 with a deviation from linearity of less than 2 %. The size of the sensitive detector area shall be optimized in order to exclude a deterioration of the integration process in the sphere and influence of speckle on the measurement. The detector is attached to the detection port of the sphere with its sensitive area forming approximately one part of the inner surface.

For shielding the detector against the direct radiation scattered onto the sensitive area by the specimen, radiation baffles shall be installed in the integrating sphere. The surfaces of these baffles shall be coated with or consisting of the same material as the inner surface of the integrating sphere. An additional diffusing window may be installed in front of the detector in order to compensate for spatial variations of the detector sensitivity.

A phase sensitive detection technique or an advanced data acquisition technique is recommended for improved detection sensitivity.

NOTE Phase sensitive detection schemes are typically operated in conjunction with a radiation chopper or another suitable technique installed into the beam path to modulate the output beam. The processing of the detector signal is performed by a lock-in amplifier that is synchronized to the modulation frequency of the radiation.

4.2.6 Specimen holder

The specimen holder shall allow for a non-destructive mounting and for a precise placement of the specimen with respect to the ports of the integrating sphere. For scanning the surface of the specimen, the holder may be equipped with a positioning system that is adapted to the desired lateral motion of the sample.

4.3 Arrangement with high sensitivity

For total scatter measurements of specimens with total scattering values below 10^{-4}, steps shall be taken to maximize the sensitivity of the arrangement. In this case, only lasers operating in a stable

TEM_{00}-mode shall be used as a radiation source. The integrating sphere shall be installed at a large enough distance from the last optical element of the beam preparation system to enable scattering from the spatial filter to be removed. To eliminate the need for neutral density filters for calibration, a dynamic range of the detection system greater than twice the reciprocal value of the minimum detectable total scattering is recommended. To decrease the contribution from Rayleigh scattering to the background noise of the measurement system, flushing of the arrangement with pure Helium gas or evacuation is recommended. Shielding the apparatus from radiation sources in the vicinity is recommended.

4.4 Preparation of specimens

The specimen shall have specified optical imaging properties that are defined by its refractive, reflective or diffractive functioning. This test method is not destructive and shall be applied to the actual part.

Wavelength, angle of incidence and polarization of the radiation as used in the test shall be in accordance with the specifications given by the manufacturer for normal use. If ranges are given for the values of these parameters, an arbitrary combination of wavelength, angle of incidence and polarization within these ranges may be chosen.

Storage, cleaning and preparation of the specimen is carried out according to directions given by the manufacturer for normal use.

In the absence of manufacturer-specified instructions, the following procedure shall be used.

The specimen shall be stored, prepared and tested in an environment with relative humidity higher than 40 % and lower than 60 %. Prior to testing, the specimen shall be kept in this testing environment in the packaging of the manufacturer for 24 h. The handling procedure of the specimen shall be optimized for a minimum exposure time of the specimen to the test environment.

The specimens shall be kept under cleanroom conditions in accordance with ISO 14644-1 as specified in Table 3 during the entire unpacking and preparation procedure without interruption. The specimen shall be handled by the non-optical surfaces only.

Table 3 — Cleanroom classes for the specimen preparation environment

Expected σ_{TS} %	Environment for specimen preparation
$\sigma_{TS} \geq 0,1$	Cleanroom better than class 7
$0,1 > \sigma_{TS} > 0,01$	Cleanroom better than class 6
$\sigma_{TS} \leq 0,01$	Cleanroom better than class 5
NOTE The cleanroom classes are defined according to ISO 14644-1.	

If contaminants are observed on the specimen or if the original packing was unsealed under undefined environmental conditions, the surface shall be cleaned. The cleaning procedure shall be documented. If the contaminants are not removable, they shall be documented by photographic and/or electronic means before testing.

5 Procedure

5.1 General

Conditions as stated in Table 3 for the specimen preparation environment also apply for the measurement system. For repeatable measurements, the specimens shall be kept under these conditions without interruption during the entire test procedure.

5.2 Alignment procedure

5.2.1 General

The alignment of the experimental arrangement is of central importance for the accuracy of the measurement.

5.2.2 Alignment of the beam

The beam shall pass through the centre of the entrance and exit port of the integrating sphere. The beam parameters shall have been measured by a beam profile measurement system. For a coarse inspection of the beam prior to the mounting of a specimen, a scattering surface (e.g. white cardboard) may be used for assessing the beam spot at the entrance and exit ports.

5.2.3 Alignment of the specimen

For the measurement of backward scattering, the specimen is attached to the exit port of the integrating sphere with the front surface pointing towards the sphere. The portion of the beam reflected by the component shall exit the entrance port of the sphere without influencing the measurement.

For the measurement of forward scattering, the specimen is attached to the entrance port of the integrating sphere with the rear surface pointing towards the sphere. The specularly reflected beam shall be aligned such that interference with the radiation source is excluded. The transmitted beam shall leave the sphere at the centre of the exit port.

For the alignment of the specimen, the angle of incidence shall be tilted slightly from the normal direction. An angle of 1,5° with respect to the normal direction shall not be exceeded for the measurement.

NOTE For integrating spheres with two circular beam ports, this implies that the incident beam deviates slightly from the centre of the beam ports, see Reference [6].

For other angles of incidence, the experimental arrangement shall be adapted to the special geometry, and the alterations shall be documented. The installation of a third beam port is allowable for the path of the radiation specularly reflected by the specimen. If a spatial scanning system is provided, the alignment conditions for the specimen shall be fulfilled for the entire scanning range.

5.3 Measurement procedure

In the first step, a calibrated power meter shall be placed at the measurement position and the beam power as well as the signal of the monitor detector shall be recorded. The power meter shall be removed and a diffuse reflectance standard shall be attached to the exit port such that its surface forms a part of the inner surface of the integrating sphere. The reading, V_c, of the detection system shall be recorded. To avoid errors caused by nonlinearities of the detection system, neutral density filters with known attenuation may be used. For the evaluation of the background noise signal, the diffuse reflectance standard shall be removed, and the signal of the unloaded sphere, V_u, shall be recorded.

In the next step, the specimen shall be attached to the port. After aligning the specimen, the reading, $V_{s,bac}$ or $V_{s,for}$, of the detection system shall be recorded for the position or scanning range provided on the specimen. The direction of scanning and the geometric scanning range on the surface of the optical component shall be documented. The scanning range shall be referred to fixed reference points on the specimen. It is acceptable to make marks at locations on the non-optical surfaces of the specimen as reference points.

If scanning of the specimen is not specified, the procedure shall be repeated for at least five different beam positions r_i on the specimen surface. For samples with low uniformity of the surface, an increased number of different beam positions, r_i, shall be measured.

6 Evaluation

6.1 Determination of the total scattering value

For a measurement without scanning the surface, the forward and backward total scatter values are determined from the measured signals, V_s and V_c, by the following Formulae (1) and (2):

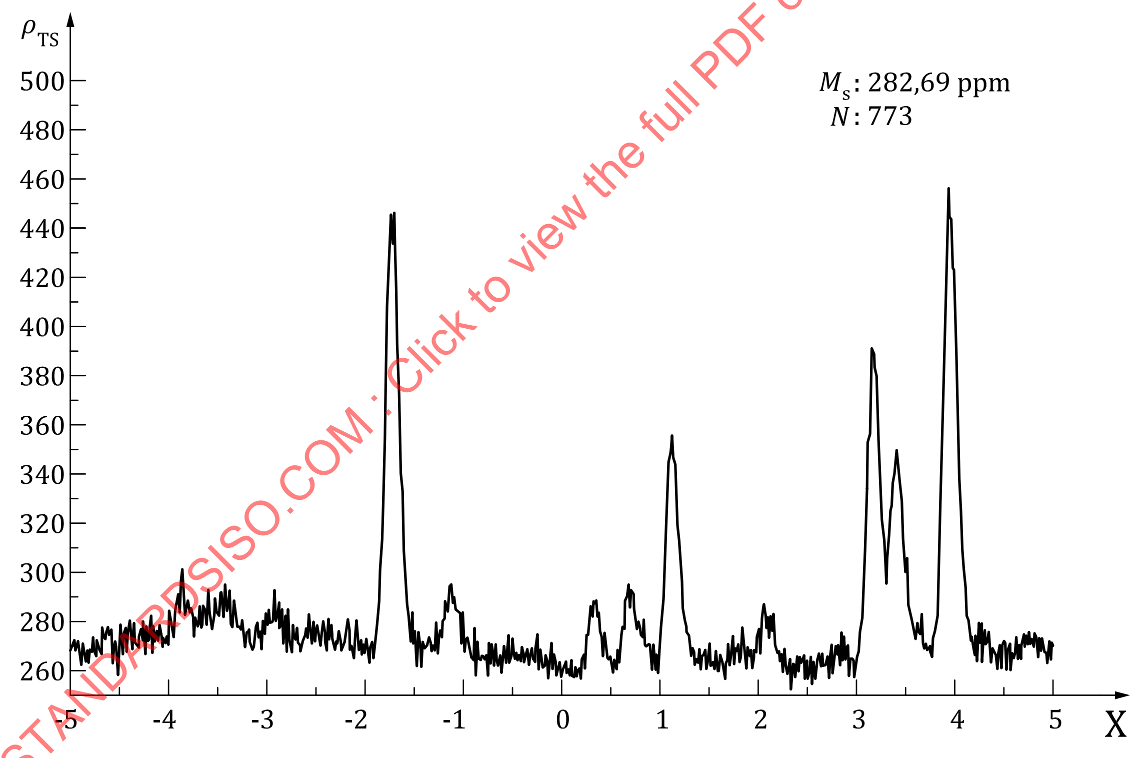
$$\tau_{TS,rs} = \frac{1}{N} \sum_{i=1}^{N} \frac{V_{s,for}(r_i)}{V_c} \quad (1)$$

$$\rho_{TS,rs} = \frac{1}{N} \sum_{i=1}^{N} \frac{V_{s,bac}(r_i)}{V_c} \quad (2)$$

NOTE 1 The subscript rs in $\tau_{TS,rs}$ and $\rho_{TS,rs}$ indicates a measurement without scanning of the specimen.

In case a calibration sample with arbitrary diffuse reflectance is used, the signal, V_c, shall be corrected in respect to the actual reflectance of the calibration sample.

A two-dimensional or three-dimensional plot (see Figure 2) shall be used for the presentation of the total scatter values measured with a scanning device.



Key

X scan position in mm

ρ_{TS} total backward scattering in ppm

M_s average total backward scattering value

Figure 2 — Graph showing the total backward scattering values recorded during a scan of a sample

The calculation of the scatter values for a scanning position, r_i, refers to the calibration signal, V_c, determined before the measurement of the specimen as given by Formulae (3) and (4):

$$\tau_{TS,sc}(r_i) = \frac{V_{s,for}(r_i)}{V_c} \tag{3}$$

$$\rho_{TS,sc}(r_i) = \frac{V_{s,bac}(r_i)}{V_c} \tag{4}$$

NOTE 2 Formulae (1), (2), (3), and (4) are valid only if the contribution of the signal V_u of the unloaded sphere to the total scatter value is not significant. Scanning of the calibration sample is advisable. The subscript sc in $\tau_{TS,sc}$ and $\rho_{TS,sc}$ indicates a measurement with scanning of the specimen.

In case a diffuse reflectance standard with arbitrary diffuse reflectance is used, the signal, V_C, shall be corrected in respect to the actual reflectance of the calibration sample. The total scatter value is determined from a statistical evaluation of the raw data $\tau_{TS,sc}(r_i)$ or $\rho_{TS,sc}(r_i)$ by plotting the number of positions with scatter values in the interval $[S, S + dS)$ as a function of S, the measured scatter value, (see Figure 3). The quantity, dS, is chosen such that a representative number of positions are located in the interval $[S, S + dS)$ at the maximum of the distribution function (see Annex C).

NOTE 3 The notation $[S, S + dS)$ indicates the scatter values in the interval S to $S + dS$, including the value S, but excluding the value $S + dS$.

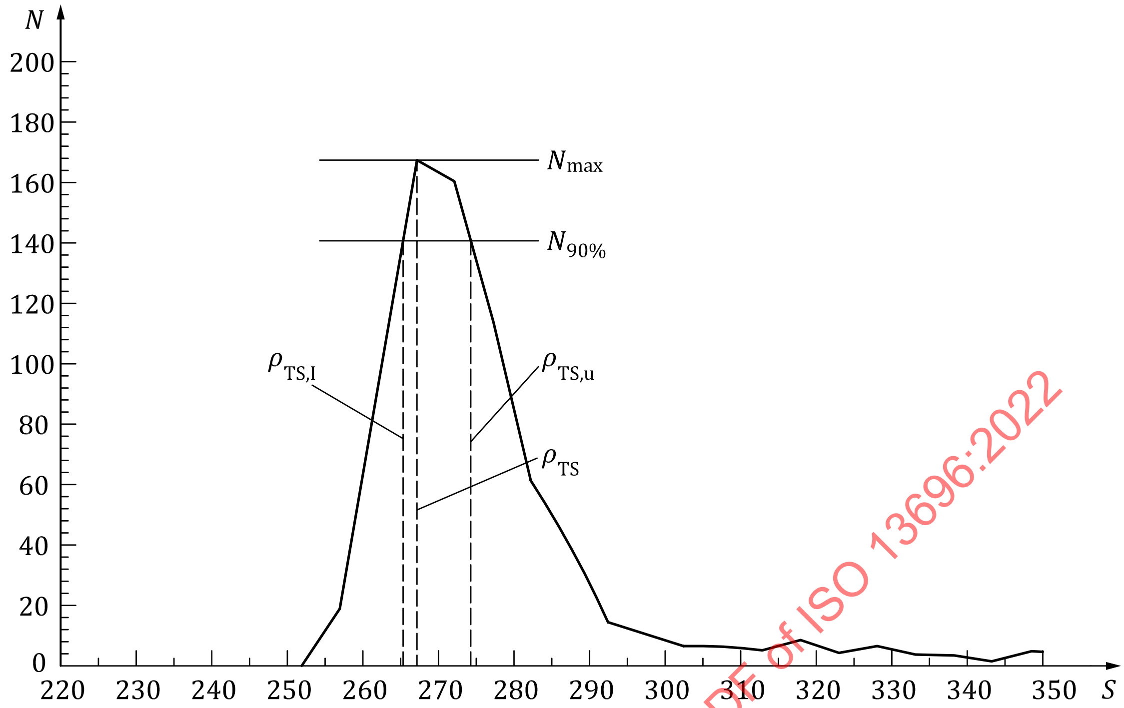
The scatter behaviour of the specimen is then represented by a set of three scatter values (see Figure 3):

— (τ_{TS} or ρ_{TS}): scatter value at the maximum of the distribution;

— ($\tau_{TS,l}$ or $\rho_{TS,l}$): lower scatter value attributed to 90 % of the distribution;

— ($\tau_{TS,u}$ or $\rho_{TS,u}$): higher scatter value attributed to 90 % of the distribution.

A more detailed statistical evaluation is optional and shall be presented in comprehensible steps. An example for a data reduction technique, which results in a single relevant scatter value, is described in Annex C. The scanning length on the specimen surface and the total number of measurement points for the scatter distribution diagram shall be documented.



Key

S scatter value

N number of sites

ρ_{TS} total backward scattering in ppm

Figure 3 — Statistical analysis of the total backward scattering values for a surface scan

If the contribution of the signal V_u of the unloaded sphere to the total scatter value is significant, a correction of the expressions with respect to V_u shall be performed. For set-ups, where Rayleigh scattering in the integrating sphere is the dominant contribution to the signal V_u, a first order correction is given by the following Formulae (5) to (8):

$$\tau_{TS,rs} = \frac{1}{N}\sum_{i=1}^{N} \frac{V_{s,for}(r_i) - (\tau_s V_u)}{V_c - 2V_u} \quad (5)$$

$$\rho_{TS,rs} = \frac{1}{N}\sum_{i=1}^{N} \frac{V_{s,bac}(r_i) - (1+\rho_s)V_u}{V_c - 2V_u} \quad (6)$$

$$\tau_{TS,sc}(r_i) = \frac{V_{s,for}(r_i) - (\tau_s V_u)}{V_c - 2V_u} \quad (7)$$

$$\rho_{TS,sc}(r_i) = \frac{V_{s,bac}(r_i) - (1+\rho_s)V_u}{V_c - 2V_u} \quad (8)$$

where

ρ_s is the spectral reflectance;

τ_s is the transmittance of the specimen.

In this approximation, the contribution of the scatter signal related to the unloaded sphere is determined from the fraction of radiation in the sphere which is transmitted through the specimen (forward scattering) or reflected back by the specimen (backward scattering). The approximation shall be applied only if the measured signals $V_{s,bac}$ or $V_{s,for}$ are at least one order of magnitude higher than

V_u. Other techniques for the background correction are applicable and shall be documented in the test report. In case a diffuse reflectance standard with arbitrary diffuse reflectance is used, Formulae (5) to (8) shall be corrected in respect to the actual reflectance, ρ_{CS}, of the calibration sample as follows:

$$\tau_{TS,rs} = \frac{1}{N}\sum_{i=1}^{N} \frac{V_{s,for}(r_i) - (\tau_s V_u)}{V_c - (1+\rho_{CS})V_u} \quad (9)$$

$$\rho_{TS,rs} = \frac{1}{N}\sum_{i=1}^{N} \frac{V_{s,bac}(r_i) - (1+\rho_s)V_u}{V_c - (1+\rho_{CS})V_u} \quad (10)$$

$$\tau_{TS,sc}(r_i) = \frac{V_{s,for}(r_i) - (\tau_s V_u)}{V_c - (1+\rho_{CS})V_u} \quad (11)$$

$$\rho_{TS,sc}(r_i) = \frac{V_{s,bac}(r_i) - (1+\rho_s)V_u}{V_c - (1+\rho_{CS})V_u} \quad (12)$$

6.2 Error budget

The error budget of the measurement shall be evaluated by considering the fluctuations of the beam parameters and the detector signal of the unloaded integrating sphere. Inaccuracies of the detector and the power monitoring system shall be included in the error budget. An example of an error budget for a total scatter measurement with a HeNe-Laser is given in Table 4. Because of the statistical nature of optical scattering phenomena, the accuracy of a scatter measurement is dependent on the properties of the individual specimen. Therefore, the error budget is restricted to the accuracy of the measurement facility.

Table 4 — Typical error budget for a total scatter measurement facility with a HeNe-Laser

Random variations	
Variation of the incident power, P_{inc}	5 %
Variation of the beam diameter, d_o	3 %
Variation of the signal processing system	2 %
Systematic errors	
Calibration of the power measurement system	3 %
Nonlinearity of the detector system	2 %
Calibration procedure, diffuse reflectance standard	5 %
Detector and signal processing noise	$0,5 \times 10^{-6}$
Signal of the unloaded sphere, V_u	$1,2 \times 10^{-6}$

NOTE 1 The values for the detection limit are given in units of the total scattering corresponding to the respective signals.

NOTE 2 The contribution of the temporal variation in the laser power, P_{inc}, to the error can be minimized by recording P_{inc} and by correlating the measured total power, P_{bac} or P_{for}, to the actual value of the incident power, P_{inc}.

7 Test report

The test report shall include the following information:

a) Information concerning the testing laboratory:

1) name and address of the testing organization;

2) date of test;

3) name of the operator of the measurement system;

4) a reference to this document, i.e. ISO 13696:2022, used as basis for the test.

b) Information on the specimen:

1) manufacturer of the specimen, part identification code, date of production;

2) description of the sample (materials, coating, polishing, diameter and thickness);

3) specifications of the manufacturer for storage and cleaning;

4) specifications of the manufacturer for normal use (spectral characteristics, wavelength, polarization, angle of incidence, purpose).

c) Information on the test:

1) equipment (laser, sphere, monitoring and detection system, and components for beam shaping);

2) parameters of the radiation source (wavelength, state of polarization, output power, spatial beam profile);

3) parameters of the sphere (diameter of the sphere and the ports, coating material, beam diameter on the specimen surface);

4) parameters of the detection system (wavelength range, linearity, sensitivity);

5) error budget (see Table 4);

6) angle of incidence;

7) angle of polarization;

8) number of test sites on the specimen surface;

9) arrangement of the test sites on the specimen surface;

10) geometrical scanning range;

11) type of calibration sample;

12) test environment.

d) Information on the result:

1) total scattering value and 90 %-points;

2) diagram for tests with surface scanning, total scattering as a function of scanning position (see Figure 2);

3) diagram for tests with surface scanning, number of sites per total scatter value (see Figure 3) with presentation of the total scattering value (maximum) and 90 %-points;

4) statistical analysis for the distribution of total scattering values (optional).

An example for a test report is given in Annex B.

Annex A
(informative)

Set-up with a Coblentz hemisphere

A.1 Principle

A further possibility of realizing an apparatus for total scattering measurement is based on the collection of scattered light using a hemispherical mirror (Coblentz sphere) which images the scattered radiation onto the detector (see Figure A.1). The hemisphere for measurements under near normal incidence has an aperture near its centre, which represents the entrance/exit port through which both the incident and specularly reflected laser beam have to pass. Both the sample and detector unit are placed in the plane of the diameter of the hemisphere such that they are as close as possible to the centre of curvature of the sphere.

The basic principle of imaging the radiation scattered by the specimen results in a lower sensitivity of the Coblentz sphere to Rayleigh scattering by the environment. Depending on the applied mirror coating, Coblentz spheres can be used for the entire wavelength range specified in this document. They are often preferred for measurements in the wavelength range below 250 nm and can be operated in the deep ultraviolet below 200 nm limited by the reflectance of the mirror coating.

NOTE It has been demonstrated that, for specimens which exhibit only backward scattering, the scatter measurement results of Coblentz spheres are comparable to measurements by Ulbricht spheres.

In the following, detailed descriptions will be given only with respect to those elements of the measurement facility and measurement procedures that differ from the description for the Ulbricht sphere; i.e. for all items not mentioned in this annex, the same definitions, descriptions and procedures apply as those outlined in Clauses 1 to 6.

A.2 Experimental set-up

A.2.1 Coblentz sphere

A Coblentz sphere is used for the collection of the radiation scattered by the sample into the backward direction. The inner surface of the hemisphere should be coated with a highly reflective metal layer or layer system. The quality of the surface finish as well as of the coating should guarantee that the irradiated portion of the specimen is imaged within the detector unit area.

The minimum possible diameter of the Coblentz sphere is subject to the requirement that all radiation scattered from the specimen into the specified angle interval from 2,0° to 85° is imaged within the detector unit area.

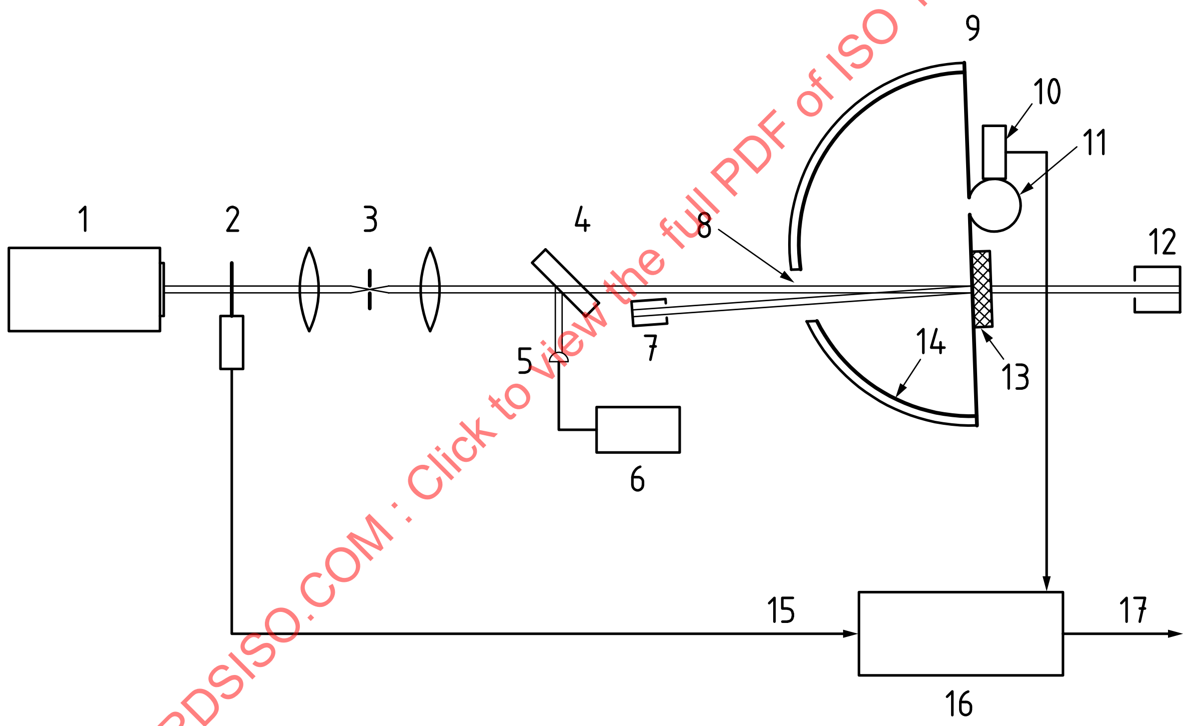
The sphere should be equipped with an entrance/exit port for the probe beam. This port is located near the centre of the mirror, directly opposite the specimen position (see Figure A.1). The size of the entrance/exit port should be realized such that the specified near-angle limit of backscattering is accomplished.

The sample holder and the detector unit should be mounted in the plane of the diameter of the hemisphere at conjugate places close to the centre of curvature. The sample holder is adjusted such that the front surface of the specimen is located exactly in the plane of the diameter of the hemisphere.

NOTE The sample and the detector can be located slightly out of the plane of the diameter of the hemisphere to optimize the imaging of the sphere and the flexibility in respect to sample geometries. As a consequence of the aberrations induced by the imaging properties of a hemisphere, deviations of the measured scatter values can appear for different beam shapes and for samples with significant volume scattering or augmented scattering into larger angles.

The detector unit consists of the detector itself and a diffuser (for example small integrating sphere or transparent diffuser) mounted in front of the detector. The diffuser is for preventing any variations of the detector signal with the angle of incidence and the position of the radiation on the detector.

The dimension of the hemispherical mirror together with that of the entrance/exit port as well as the effective area of the detector unit (i.e. size of the entrance port of the small integrating sphere) are chosen such that full imaging of all radiation scattered onto the detector unit area will be guaranteed in the specified range of collected backscattering angles (2,0° to 85°).



Key

1 radiation source
2 chopper
3 spatial filter
4 beam splitter
5 power detector
6 power meter
7 beam stop
8 entrance port
9 Coblentz sphere
10 detector
11 integrating sphere
12 beam stop
13 sample
14 coating
15 chopper signal
16 lock-in amplifier (optional)
17 detector signal, V_s

Figure A.1 — Schematic arrangement for the measurement of total scattering with a Coblentz sphere (configuration in backward scattering with phase sensitive detection scheme)

A.2.2 Calibration

For calibration, a diffuse reflectance standard is mounted in the sample holder.

NOTE Take care, in case a volume diffuser is used as diffuse reflectance standard. Volume scattering materials can cause a broadening of the diffuse emission profile of the diffuse reflectance standard.

A.2.3 Alignment of the specimen

The specimen is positioned in the sample holder with the front surface pointing towards the hemisphere. The specularly reflected beam shall exit the entrance/exit port without influencing the measurement.

Annex B
(informative)

Example of test report

Radiation scattered by optical components (ISO 13696)

<u>**Testing institute**</u>

Testing institute:	Xaa Xaaaaa Xaaa, Xaaaaa xaa XX Xaaaa XXX, X-XXXX Xaaaaa XX
Date/Tester:	28/02/1994/ X. Xaaaaaaaa

<u>**Specimen**</u>

Type of specimen:	HR at 633 nm on BK7 glass, laser grade polishing, diameter 25 mm, thickness 5 mm, polarization not specified by vendor
Manufacturer:	Xaa Xaaaaa XaaaXaaaaa xaa XX Xaaaa XXX, X-XXXX Xaaaaa XX
Strorage, cleaning:	No special requirements
Specification:	High reflecting coating,
	R > 99,8 %, T < 0,2 % at 633 nm
	0 rad angle of incidence
	Coating for HeNe cavity mirrors
Part identification,	
date of production:	Coating Run No. 15AC of 1992-08-31

<u>**Test specification**</u>

Set-up with HeNe-laser (type, manufacturer), integrating sphere (type, manufacturer), photodiode monitoring, phase sensitive detection system (type, manufacturer), photomultiplier (type, manufacturer) and telescopic arrangement for beam preparation.

Laser parameters:

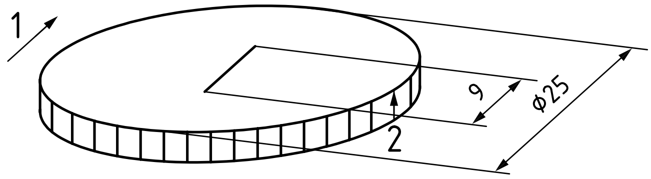
Wavelength	633 nm
State of polarization	linear
Angle of polarization	0°
Output power	3,5 mW
Beam profile in target plane	TEM_{00}

Integrating sphere:

Diameter	250 mm
Port diameter	12 mm

Coating material	barium sulfate
Range of acceptance angle	1,38° to 88°
Beam diameter, d_σ, on the specimen surface	400 µm
Detection system:	
Wavelength range	450 nm to 700 nm
Dynamic range	10^5
Linearity error	<5 %
Sensitivity	1 V/µW
Error budget:	
Random variations	
Incident power, P_{inc}	5 %
Variation of the beam diameter, d_σ	3 %
Variation of the signal processing system	2 %
Systematic errors	
Calibration of the power measurement system	3 %
Nonlinearity of the detector system	2 %
Calibration procedure, 100 % standard	5 %
Detector and signal processing noise	$0,3 \times 10^{-6}$
Signal of the unloaded sphere, V_u	$1,5 \times 10^{-6}$
Signal of the unloaded sphere, V_u, (Helium)	$0,6 \times 10^{-6}$
Test parameters:	
Angle of incidence	<1,5°
Angle of polarization	0°
Number of sites on the specimen	1 024
Arrangement of test sites	scan (see Figure B.1)
Scanning range	9 mm
Calibration sample	diffuse reflectance standard, R = 100 %
Test environment	Flow bench, cleanroom, class <5, see ISO 14644-1

Dimensions in millimetres



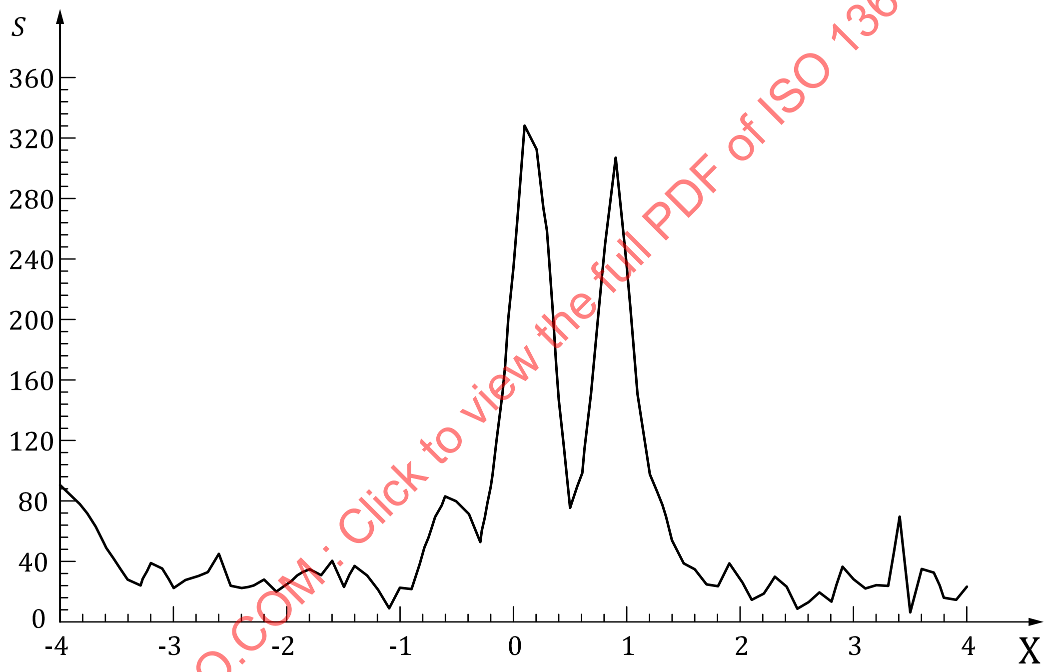
Key

1 scan direction

2 mark

Figure B.1 — Arrangement of the test sites on the specimen

<u>**Test results**</u>

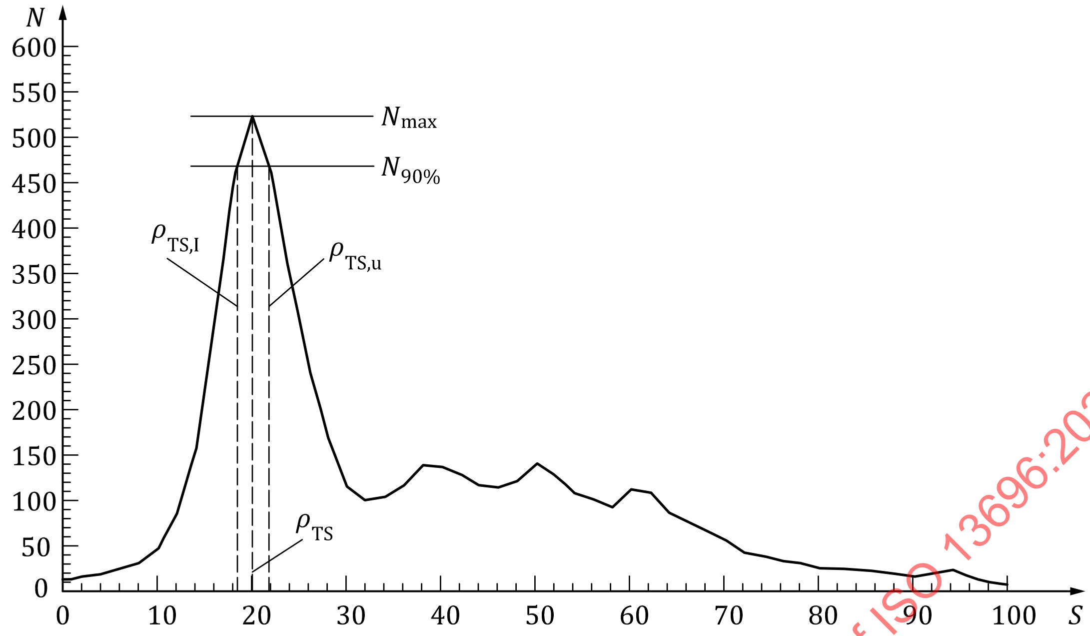


Key

X scan position in mm

S scatter value

Figure B.2 — Total backward scattering values as a function of the scanning position



Key

S	scatter value
N	number of sites
ρ_{TS}	total backward scattering in ppm

Figure B.3 — Statistical analysis of the total backward scattering values for the scan of Figure B.2

Total scattering values (see Figures B.2 and B.3)

Backward scattering, ρ_{TS}	20,1 ppm
Lower 90 %-point, $\rho_{TS,l}$	18,5 ppm
Upper 90 %-point, $\rho_{TS,u}$	21,9 ppm

Comment

Scratches on the specimen surface in the middle of the scanning range.

Annex C
(informative)

Statistical evaluation example

C.1 General

In this annex, a data reduction technique for the determination of a value for the quantity, dS (see 6.1), is described and illustrated by measured data.

C.2 Data reduction algorithm

The result of a total scattering measurement with scanning of the sample position is a number N exceeding typically several hundreds of scatter values $\tau_{TS,sc}(r_i)$ or $\rho_{TS,sc}(r_i)$ measured at the locations r_i (see Figure C.1). In order to estimate the optimum quantity, dS, the following iterative procedure may be applied.

a) Calculate the arithmetic mean value, M_s, of the scatter data by Formula (C.1) (in the case of backward scattering):

$$M_s = \frac{1}{N}\sum_{i=1}^{N} \rho_{TS,sc}(r_i) \quad (C.1)$$

b) Calculate the standard deviation, σ_s, of the scatter values by Formula (C.2):

$$\sigma_s = \sqrt{\frac{1}{N-1}\sum_{i=1}^{N}\left[M_s - \rho_{TS,sc}(r_i)\right]^2} \quad (C.2)$$

c) Select the scatter values $\tau_{TS,sc}(r_i)$ or $\rho_{TS,sc}(r_i)$ which are within the interval (M_s - 2σ_s, M_s + 2σ_s) and iterate the calculation according to steps a) and b) until the number of selected data points does not decrease further, or the relative variation of the standard deviation, σ_s, is below a factor 10^{-4}.

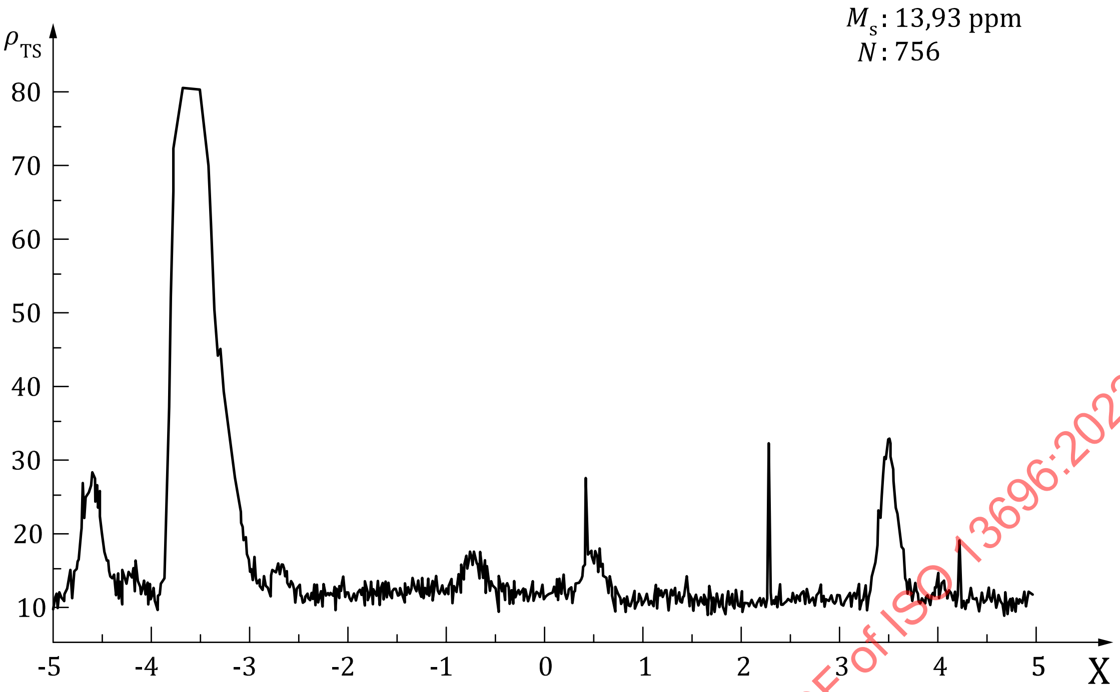
d) Using the attained standard deviation as a value for the quantity, dS, plot the scatter distribution function according to 6.1 and determine the representative scatter values (τ_{TS} or ρ_{TS}), ($\tau_{TS,l}$ or $\rho_{TS,l}$) and ($\tau_{TS,u}$ or $\rho_{TS,u}$).

NOTE The calculated standard deviation, σ_s, of a measured data set is generally not equal to the standard deviation of the distribution function constructed according to 6.1. In most cases, the standard deviation of the intrinsic contribution to the scattering can be determined by a least squares fit of a Gaussian distribution function to the distribution function which is constructed with the quantity, dS, determined by the data reduction algorithm.

C.3 Data reduction example

In Figure C.1, an arbitrarily selected scatter measurement scan is shown for a multilayer coating of TiO_2/SiO_2 on an optically polished substrate. Besides peaks in the scatter map, which are attributed to localized defects on the surface or in the coating, a constant scatter level is observed for the intrinsic scatter behaviour of the surface.

a) The importance of the quantity, dS, for the evaluation procedure is illustrated in Figure C.2, where scatter distribution functions are plotted for different values of dS. Meanwhile, the arithmetic mean value, M_s, of the scan is influenced only in the range of a few percents, the standard deviation, σ_s, and consequently, the parameters ($\tau_{TS,l}$ or $\rho_{TS,l}$) and ($\tau_{TS,u}$ or $\rho_{TS,u}$) are drastically affected by a variation of the quantity, dS.

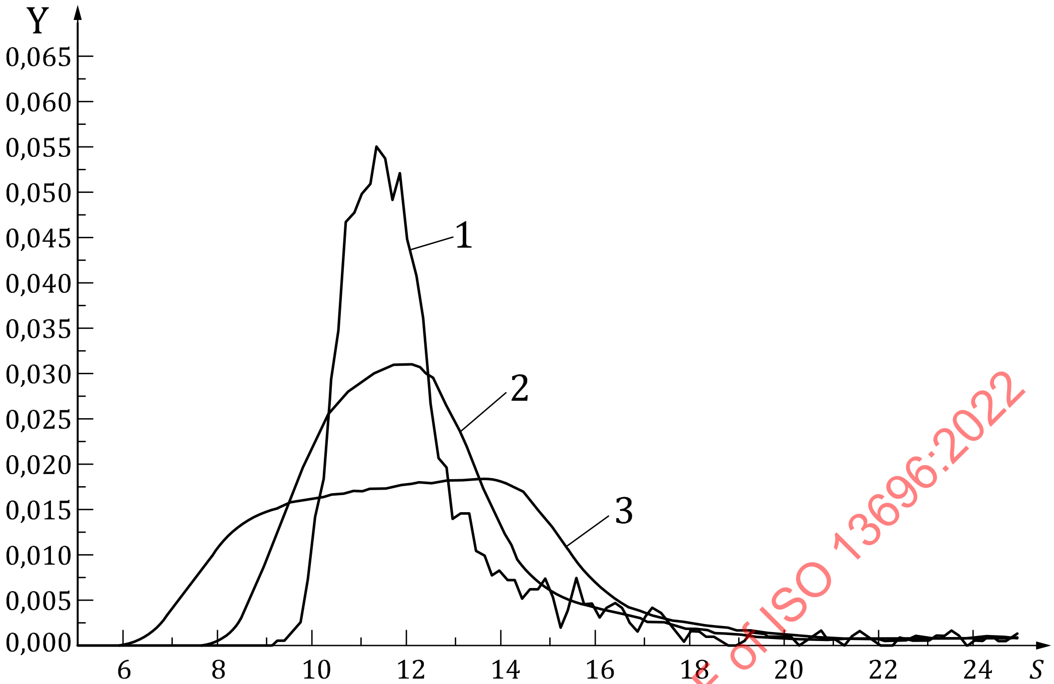


Key

X scan position in mm

ρ_{TS} total backward scattering in ppm

Figure C.1 — Total backward scattering values as a function of the scanning position



Key

S	total backward scattering in ppm	Y	probability	
1	d*S* = 0,4	M_s =	11,37	σ_s = 0,91 ppm
2	d*S* = 4,0	M_s =	11,59	σ_s = 1,72 ppm
3	d*S* = 7,7	M_s =	11,84	σ_s = 2,97 ppm

NOTE The frequency of the measured scatter values is given in calibrated units of probability.

Figure C.2 — Distribution diagrams of the measured backward scatter values for selected values of the quantity, d*S*

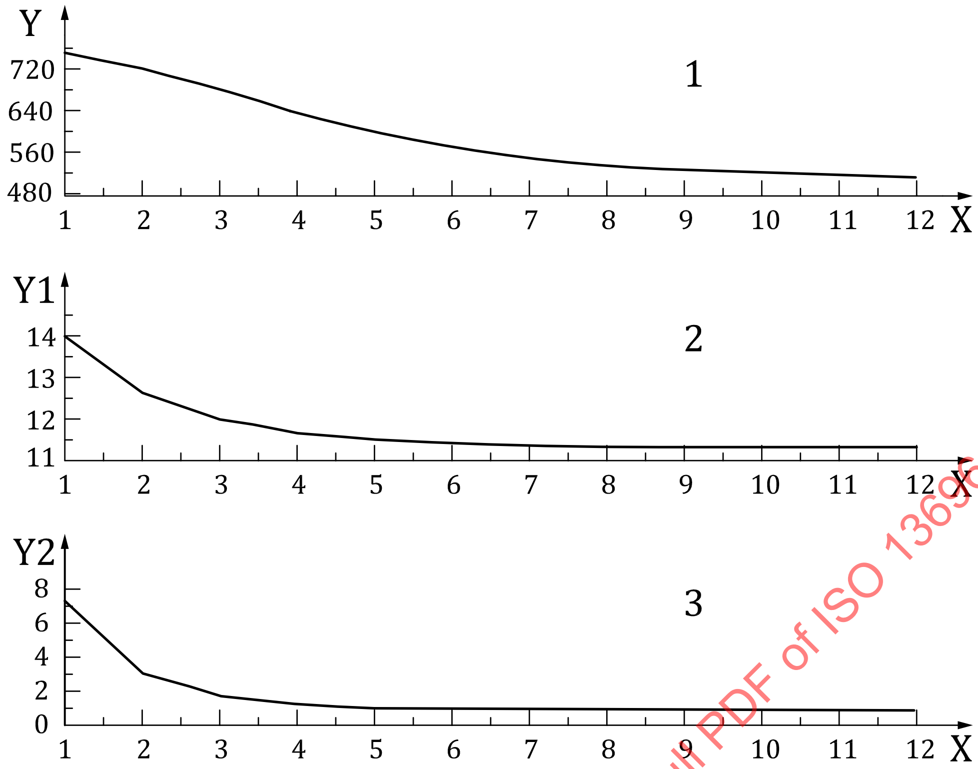
The development of the specific parameters M_s, σ_s and the number of selected data points during the data reduction algorithm, is depicted in Figure C.3. After the steps a) to c) have been executed several times, the parameters stabilize to saturation values, which are characteristic of the intrinsic scatter level of the sample.

Using the standard deviation of the data reduction algorithm as a value for the quantity, d*S*, the optimized scatter distribution can be plotted (see Figure C.4). From the diagram, the parameters can be deduced (see 6.1):

ρ_{TS} = 11,36 ppm

$\rho_{TS,l}$ = 10,72 ppm

$\rho_{TS,u}$ = 11,68 ppm



Key

X iteration

Y count

Y1 mean in ppm

Y2 standard deviation in ppm

1 data reduction 32 % from 756 to 514 points

2 mean reduced by 18,8 % from 13,93 ppm to 11,31 ppm

3 standard deviation reduced by 91,7 % from 7,7 ppm to 0,64 ppm

NOTE The data are depicted as a function of the iterative step number and (are) connected by lines to guide the eye.

Figure C.3 — Development of the specific parameters M_s, σ_s and the number of selected data points during the data reduction algorithm