

# INTERNATIONAL STANDARD

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## Optics and optical instruments — Test methods for radiation scattered by optical components

*Optique et instruments d'optique — Méthodes d'essai du rayonnement  
diffusé par les composants optiques*



Reference number  
ISO 13696:2002(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.

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.

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

International Standard ISO 13696 was prepared by Technical Committee ISO/TC 172, *Optics and optical instruments*, Subcommittee SC 9, *Electro-optical systems*.

Annexes A to D of this International Standard are for information only.

## 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 International Standard describes a testing procedure for the corresponding quantity, the total scattering (TS) value, which is defined by the measured values of backward scattering and forward scattering. The measurement principle described in this International Standard is based on an Ulbricht sphere as the integrating element for scattered radiation. An alternative apparatus with a Coblentz hemisphere, which is also frequently employed for collecting scattered light, is described in annex A. Currently, advanced studies on the comparability and the limitations of both light collecting elements are being performed (e.g. round robin tests, EUREKA-project EUROLASER: CHOCLAB).

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# Optics and optical instruments — Test methods for radiation scattered by optical components

## 1 Scope

This International Standard 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 and backward scattering to the total scattering of an optical component.

This International Standard applies to coated and uncoated optical components with optical surfaces that have a radius of curvature of more than 10 m. The wavelength range includes the ultraviolet, the visible and the infrared spectral regions.

## 2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard 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 11145, *Optics and optical instruments — Lasers and laser-related equipment — Vocabulary and symbols*

ISO 14644-1:1999, *Cleanrooms and associated controlled environments — Part 1: Classification of air cleanliness*

## 3 Terms, definitions and symbols

### 3.1 Terms and definitions

For the purposes of this International Standard, the terms and definitions given in ISO 11145 and the following apply.

#### 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 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 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 into the forward or the backward halfspace or both to the power of the incident radiation

NOTE The halfspace in which the scattering is measured should be clearly stated.

### 3.1.7

#### **diffuse reflectance standard**

diffuse reflector with known total reflectance

NOTE 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 [5] in the Bibliography.)

### 3.1.8

#### **range of acceptance angle**

range from the minimum to the maximum angle with respect to the reflected or transmitted beam that can be collected by the integrating element

### 3.1.9

#### **angle of polarization**

$\gamma$

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

NOTE 1 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 The angle of polarization,  $\gamma$ , is identical to the azimuth,  $\phi$  (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
$\lambda$	Wavelength	nm
$\alpha$	Angle of incidence	degrees
$\gamma$	Angle of polarization	degrees
$d_G$	Beam diameter on the surface of the specimen	mm
$P_{\text{inc}}$	Power of the incident radiation	W
$P_{\text{bac}}$	Total power, backward scattered radiation	W
$P_{\text{for}}$	Total power, forward scattered radiation	W
$S_{\text{bac}}$	Backward scattering	
$S_{\text{for}}$	Forward scattering	
$V_{\text{s,bac}}$	Detector signal for the specimen, backward scattering	V
$V_{\text{s,for}}$	Detector signal for the specimen, forward scattering	V
$V_{\text{c}}$	Detector signal, diffuse reflectance standard	V
$V_{\text{u}}$	Detector signal, test ports open	V
$\tau_{\text{s}}$	Transmittance of specimen at wavelength, $\lambda$	
$\rho_{\text{s}}$	Reflectance of specimen at wavelength, $\lambda$	
$r_i$	Sample position	mm
$N$	Number of test sites per surface	

## 4 Test method

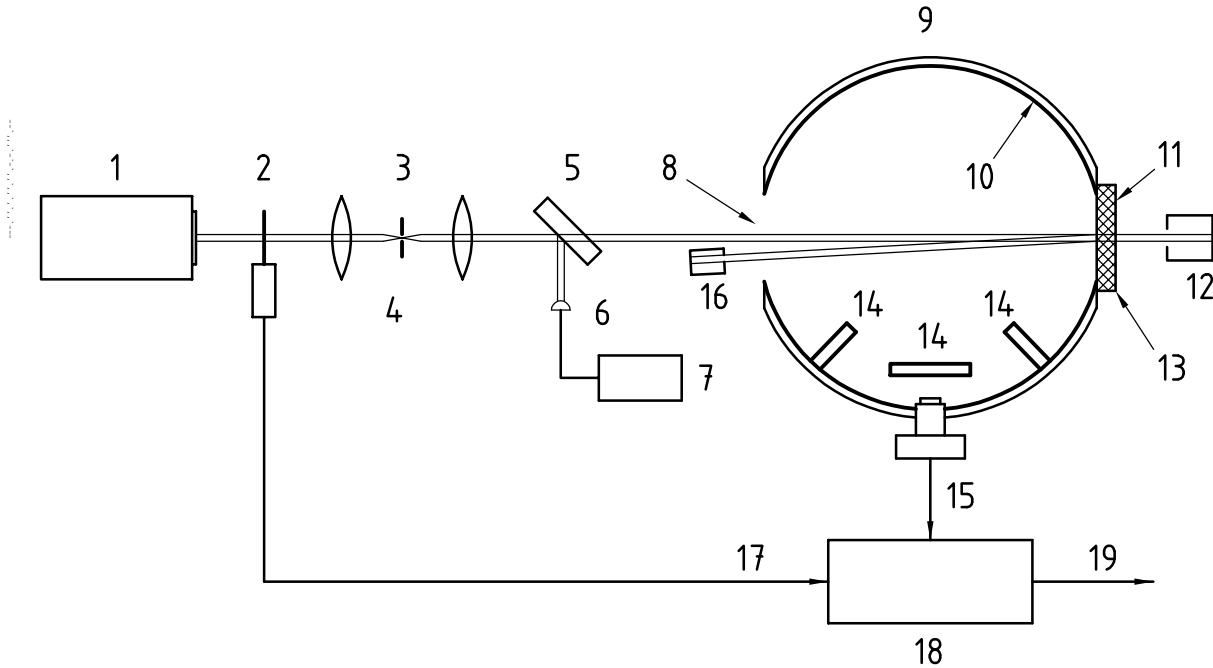
### 4.1 Principle

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 employed. 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, that 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 employed 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 | 11 Exit port              |
| 2 Chopper          | 12 Beam stop              |
| 3 Spatial filter   | 13 Sample                 |
| 4 Telescope        | 14 Radiation baffles      |
| 5 Beam splitter    | 15 Detector, diffuser     |
| 6 Power detector   | 16 Beam stop              |
| 7 Power meter      | 17 Chopper signal         |
| 8 Entrance port    | 18 Lock-in amplifier      |
| 9 Ulbricht sphere  | 19 Detector signal, $V_s$ |
| 10 Coating         |                           |

**Figure 1 — Schematic arrangement for the measurement of total scattering (configuration for backward scattering)**

**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 involving the wavelength dependence of scattering, different conventional radiation sources may be used in conjunction with spectral filters or monochromators. Different types of discharge, arc or tungsten lamps are suitable for wavelength-resolved total scatter measurements.

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 monitor detector shall be calibrated to the power at the sample surface 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,  $d_\sigma$ , 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 diameter by a factor of 2,5.

NOTE 1 The behaviour of the measured total scatter value may 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,  $d_{\sigma}$ , by a factor of three. For measurement systems with a laser as the radiation source, a TEM<sub>00</sub>-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 unpolarized 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.

NOTE 2 Beam deflection mirrors and 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 employed to suppress backscattering into the integrating sphere.

NOTE 3 An efficient beam dump may 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 employed 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. Selected materials suitable for this coating and the corresponding spectral ranges are listed in Table 2.

**Table 2 — Selected materials for coating of the inner surface of the integrating sphere**

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 beam diameter,  $d_{\sigma}$ , 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 exit and entrance port and the detector 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 The determination of the minimum size of the integrating sphere originates from the beam diameter,  $d_{\sigma}$ , at the beam ports of the Ulbricht sphere. The minimum diameter of the entrance port 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 approximately 72 times the beam diameter,  $d_{\sigma}$ .)

For measurement systems with radiation sources other than lasers or special measurement conditions, the beam diameter,  $d_0$ , 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 ISO 13696 below a wavelength of 250 nm. The amount of radiation scattered at a discontinuity is a function of both the discontinuity dimensions and the wavelength of the radiation. In practice, scattering becomes less important at longer wavelengths.

As an alternative, a Coblenz half-sphere with an appropriate reflecting surface may be employed. 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 employed 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 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 is recommended for improved detection sensitivity. A radiation chopper shall be installed into the beam path to modulate the output beam of the radiation source. The processing of the detector signal is performed by a lock-in amplifier that is synchronized to the radiation chopper.

#### 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<sub>00</sub>-mode shall be employed as a radiation source. The integrating sphere shall be installed at a large enough distance from the last refractive 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 in the range of 40 % to 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 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 TS %	Environment for specimen preparation
$TS > 0,1$	Cleanroom better than class 7
$0,1 \geq TS > 0,01$	Cleanroom better than class 6
$TS \leq 0,01$	Cleanroom better than class 5
NOTE	The cleanroom classes are defined according to ISO 14644.

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 be measured periodically by a beam profile measurement system. For a coarse inspection of the beam prior to the mounting of a specimen, a white target may be employed 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 hole of the sphere without influencing the measurement.

For the measurement of forward scattering, the specimen is attached to the entrance hole 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 center of the beam ports. (See reference [5] in the Bibliography.)

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

After aligning the specimen and tuning the phase sensitive detection system to maximum output signal, 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.

In the next step, the specimen shall be removed and a diffuse reflectance standard shall be attached to the exit port such that the target 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 non-linearities of the detection system, neutral density filters with known attenuation factors may be employed.

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.

For the evaluation of the background noise signal, the target shall be removed, and the signal of the unloaded sphere  $V_u$  shall be recorded.

## 6 Evaluation

### 6.1 Determination of the total scattering value

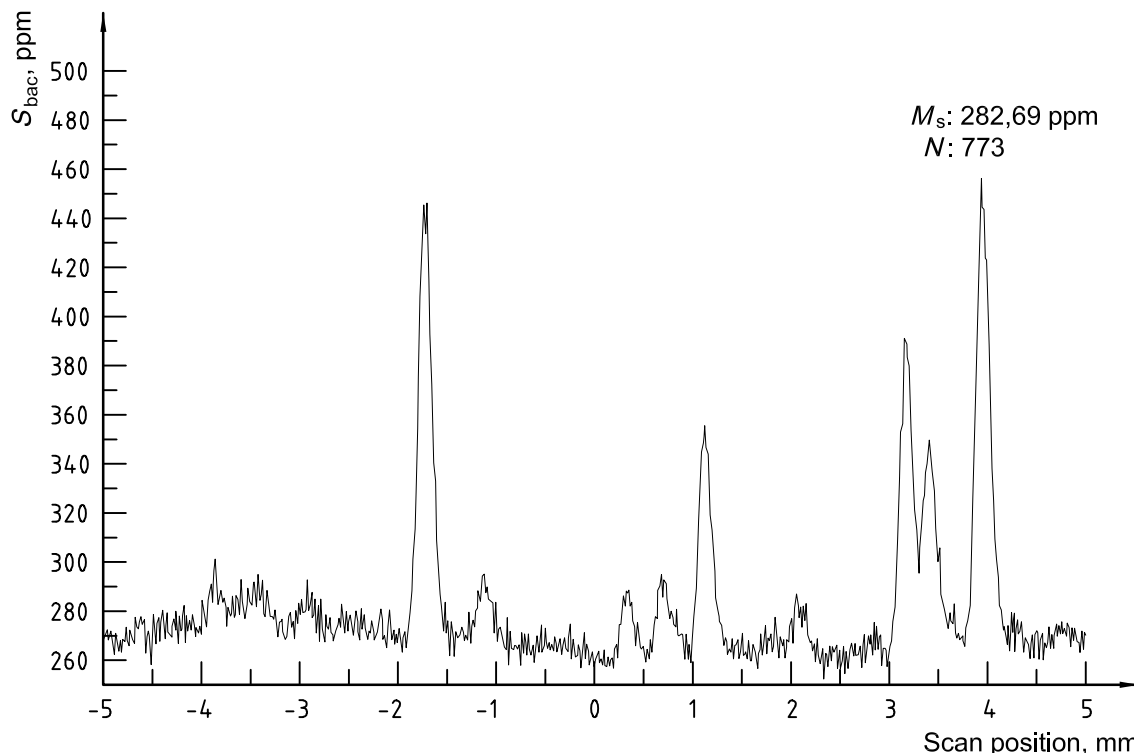
For a measurement without scanning the surface, the forward and backward total scatter values are determined from the measured signals  $V_s$  and  $V_c$  by the following expressions:

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

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

NOTE 1 The signal  $V_c(r_i)$  in equations (1) and (2) is the detector reading of the calibration sample after the position  $r_i$  of the specimen has been measured. The subscript  $r_s$  in  $S_{for,rs}$  and  $S_{bac,rs}$  indicates a measurement without scanning of the specimen.

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.



**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 after measurement of the specimen:

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

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

NOTE 2 Equations (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. In this case, the calibration signal  $V_c$  is given by the average of the measured calibration signals  $V_c(r_i)$ . The subscript  $sc$  in  $S_{for,sc}$  and  $S_{bac,sc}$  indicates a measurement with scanning of the specimen.

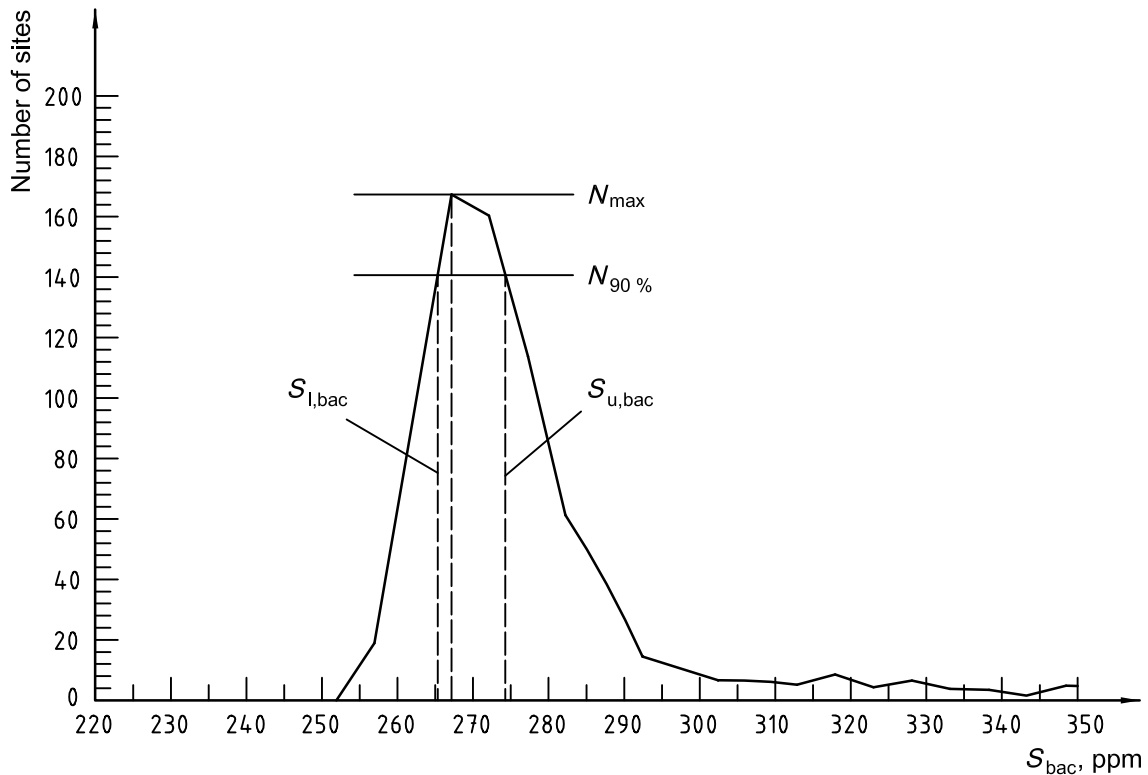
The total scatter value is determined from a statistical evaluation of the raw data  $S_{for,sc}(r_i)$  or  $S_{bac,sc}(r_i)$  by plotting the number of positions with scatter values in the interval  $(S, S + dS)$  as a function of  $S$  (see Figure 3). The quantity  $dS$  is chosen such that a representative 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):

- ( $S_{for}$  or  $S_{bac}$ ): scatter value at the maximum of the distribution;
- ( $S_{l,for}$  or  $S_{l,bac}$ ): lower scatter value attributed to 90 % of the distribution;
- ( $S_{u,for}$  or  $S_{u,bac}$ ): 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.



**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 correction is given by the following expressions:

$$S_{for} = \frac{1}{N} \sum_{i=1}^N \frac{V_{s,for}(r_i) - (s V_u)}{V_c(r_i) - V_u} \tag{5}$$

$$S_{bac} = \frac{1}{N} \sum_{i=1}^N \frac{V_{s,bac}(r_i) - (1 + s) V_u}{V_c(r_i) - V_u} \tag{6}$$

$$S_{for}(r_i) = \frac{V_{s,for}(r_i) - (s V_u)}{V_c - V_u} \tag{7}$$



$$S_{\text{bac}}(r_i) = \frac{V_{\text{s,bac}}(r_i) - (1 + \rho_s) V_u}{V_c - V_u} \quad (8)$$

where

$\rho_s$  is the spectral reflectance;

$\tau_s$  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  $V_{\text{s,bac}}$  or  $V_{\text{s,for}}$  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.

## 6.2 Error budget

The error budget of the measurement shall be evaluated by considering the fluctuations of 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**

<b>Random variations</b>	
Variation of the incident power, $P_{\text{inc}}$	5 %
Variation of the beam diameter, $d_\sigma$	3 %
Variation of the signal processing system	2 %
<b>Systematic errors</b>	
Calibration of the power measurement system	3 %
Non-linearity of the detector system	2 %
Calibration procedure, diffuse reflecting 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, $P_{\text{inc}}$ , to the error may be minimized by recording $P_{\text{inc}}$ and by correlating the measured total power, $P_{\text{bac}}$ or $P_{\text{for}}$ , to the actual value of the incident power, $P_{\text{inc}}$ .	

## 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) references to the International Standards 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 sphere

#### A.1 Principle

A further possibility of realizing an apparatus for backward 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 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. Also, Coblentz spheres can be operated in an extended spectral range in the ultraviolet below 200 nm.

**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 of this International Standard.

#### A.2 Experimental set-up

##### A.2.1 Coblentz sphere

A Coblentz sphere is employed 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 aluminium layer. 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 aluminium coating is suitable for employing the hemispherical mirror in a wide spectral range from the UV to the IR spectral region ( $\leq 200$  nm to  $\geq 10,6$   $\mu\text{m}$ ).

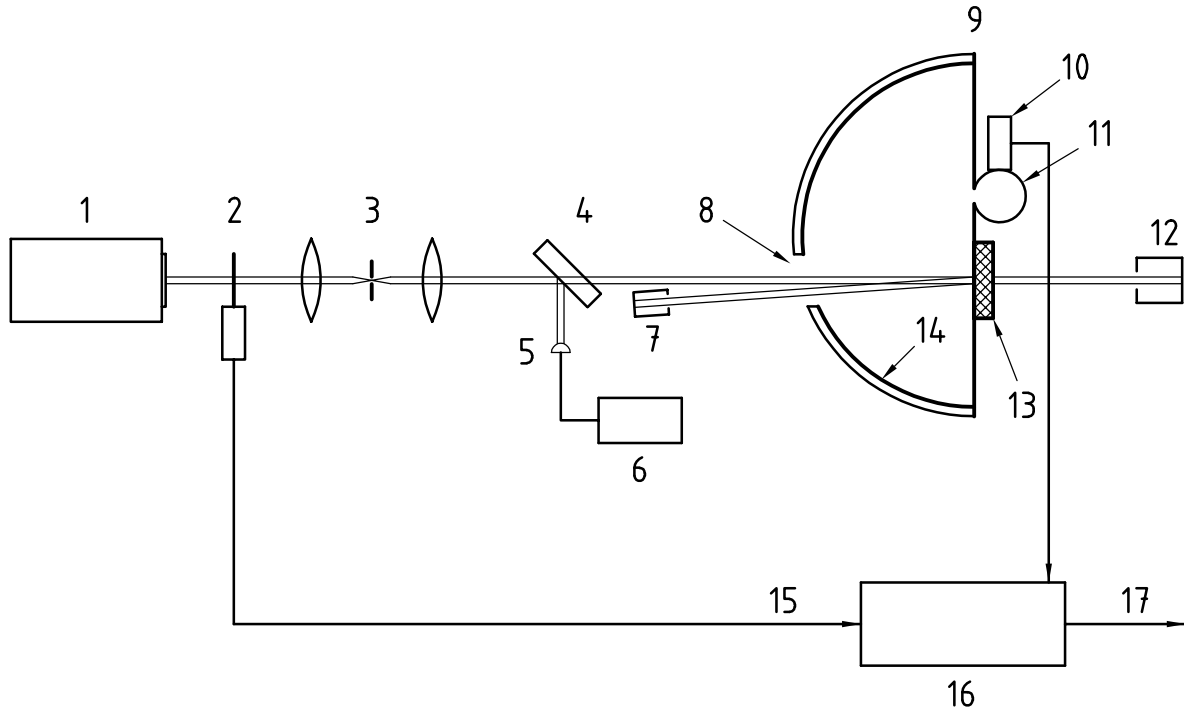
The minimum possible diameter of the Coblentz sphere is subject to the requirement that all radiation scattered from the specimen into the specified angle interval from  $2,0^\circ$  to  $85^\circ$  is imaged within the detector unit area.

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.

The detector unit consists of the detector itself and a diffuser (small integrating sphere recommended) mounted in front of the detector. The diffuser is for preventing any variations of the detector signal with the position of the image of scattered radiation.

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 in the specified range of collected backscattering angles ( $2,0^\circ$  to  $85^\circ$ ) full imaging of all radiation scattered onto the detector unit area will be guaranteed.



**Key**

- |                    |                           |
|--------------------|---------------------------|
| 1 Radiation source | 10 Detector               |
| 2 Chopper          | 11 Integrating sphere     |
| 3 Spatial filter   | 12 Beam stop              |
| 4 Beam splitter    | 13 Sample                 |
| 5 Power detector   | 14 Coating                |
| 6 Power meter      | 15 Chopper signal         |
| 7 Beam stop        | 16 Lock-in amplifier      |
| 8 Entrance port    | 17 Detector signal, $V_s$ |
| 9 Coblentz sphere  |                           |

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

**A.2.2 Alignment of the specimen**

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

**A.2.3 Calibration**

For calibration, the specimen is replaced in the sample holder by a 100 % diffuse reflectance standard.

## Annex B (informative)

### Example of test report

#### Radiation scattered by optical components (ISO 13696)

##### Testing institute

Testing institute: Xaa Xaaaaa Xaaa, Xaaaaa xaa XX Xaaaa XXX, X-XXXX Xaaaaa XX

Date/Tester: 28/02/1994/ X. Xaaaaaaaa

##### Specimen

Type of specimen: Actual specimen, HR at 633 nm on BK7 glass, laser grade polishing, diameter 25 mm, thickness 5 mm

Manufacturer: Xaa Xaaaaa XaaaXaaaaa xaa XX Xaaaa XXX, X-XXXX Xaaaaa XX

Storage, 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

##### Test specification

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:**

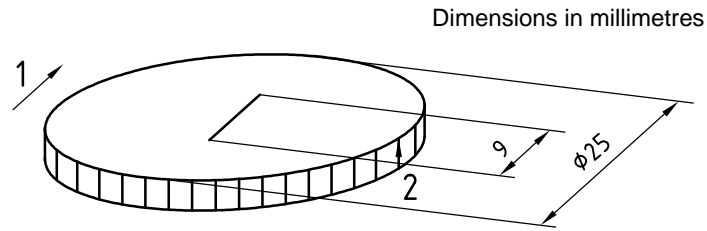
Wavelength	633 nm
State of polarization	linear
Output power	3,5 mW
Beam profile in target plane	TEM <sub>00</sub>

##### **Integrating sphere:**

Diameter	250 mm
----------	--------

## ISO 13696:2002(E)

Port diameter	12 mm
Coating material	barium sulfate
Range of acceptance angle	1,38° to 88°
Beam diameter, $d_{\sigma}$ , on the specimen surface	400 $\mu\text{m}$
<b>Detection system:</b>	
Wavelength range	450 nm to 700 nm
Dynamic range	$10^5$
Linearity error	< 5 %
Sensitivity	1 V/ $\mu\text{W}$
<b>Error budget:</b>	
Random variations	
Incident power, $P_{\text{inc}}$	5 %
Variation of the beam diameter, $d_{\sigma}$	3 %
Variation of the signal processing system	2 %
Systematic errors	
Calibration of the power measurement system	3 %
Non-linearity 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, $V_{\text{u}}$	$1,5 \times 10^{-6}$
Signal of the unloaded sphere, $V_{\text{u}}$ , (Helium)	$0,6 \times 10^{-6}$
<b>Test parameters:</b>	
Angle of incidence	< 1,5°
Angle of polarization	0°
Number of sites on the specimen	1024
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, ISO 14644-1

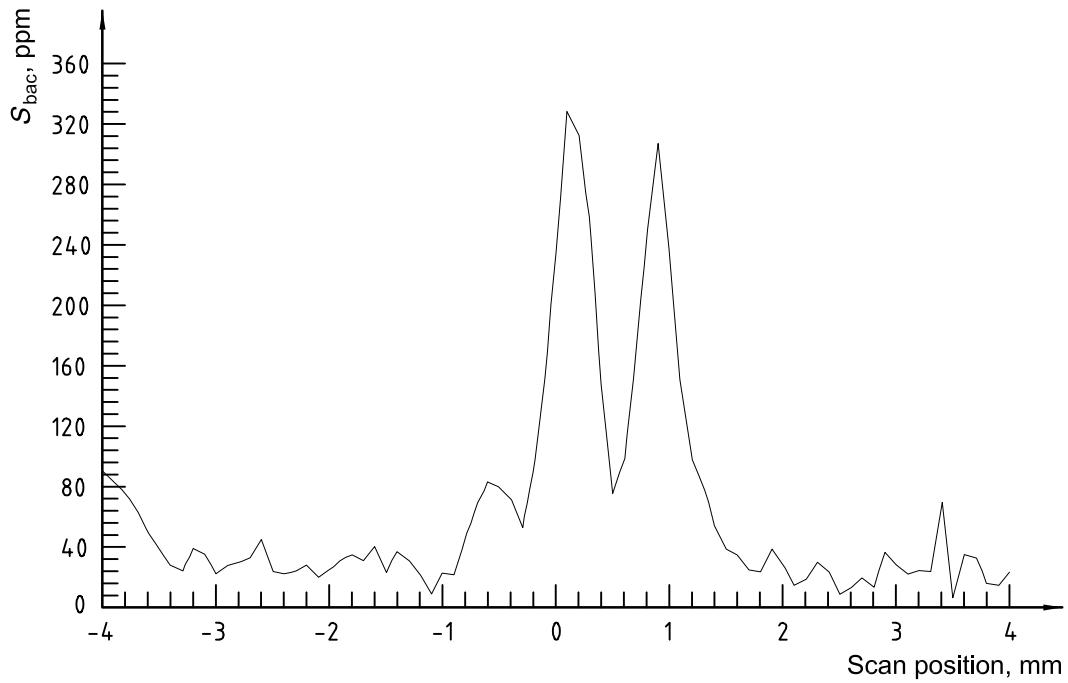


**Key**

- 1 Scan direction
- 2 Mark

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

**Test results**



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

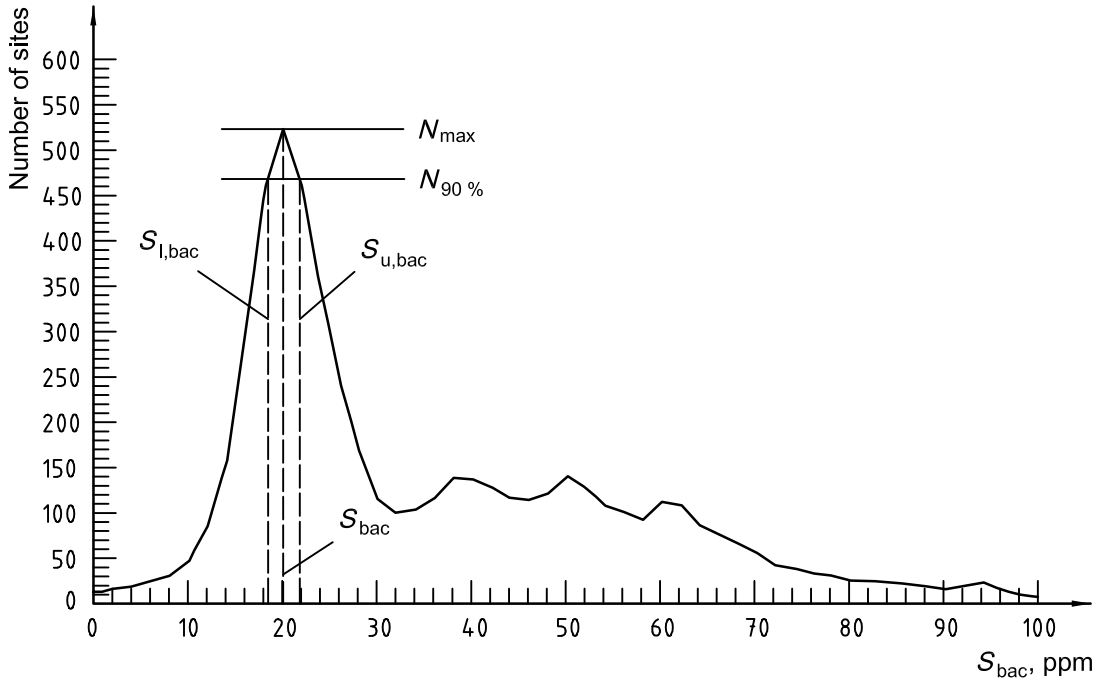


Figure B.3 — Statistical analysis of the total backward scattering values for the scan of Figure B.2

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

Backward scattering $S_{bac}$	20,1 ppm
Lower 90 %-point	18,0 ppm
Upper 90 %-point	22,6 ppm

**Comment**

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

.....



## Annex C (informative)

### Statistical evaluation example

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.1 Data reduction algorithm

The result of a total scattering measurement with scanning of the sample position is a number  $N$  of scatter value  $S_{\text{for,sc}}(r_i)$  or  $S_{\text{bac,sc}}(r_i)$  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 the equation (in the case of backward scattering)

$$M_s = \frac{1}{N} \sum_{i=1}^N S_{\text{bac,sc}}(r_i) \quad (\text{C1})$$

- b) Calculate the standard deviation  $\sigma_s$  of the scatter values by the equation

$$s = \sqrt{\frac{1}{N-1} \sum_{i=1}^N (M_s - S_{\text{bac,sc}}(r_i))^2} \quad (\text{C2})$$

- c) Select the scatter values  $S_{\text{for,sc}}(r_i)$  or  $S_{\text{bac,sc}}(r_i)$  which are within the interval  $(M_s - 2\sigma_s, M_s + 2\sigma_s)$  and restart with step 1 until the number of selected data points does not decrease further, or the relative variation of the standard deviation,  $\sigma_s$ , is below a factor  $10^{-4}$ .
- d) Using the standard deviation as a value for the quantity  $dS$ , plot the scatter distribution function according to 6.1 and determine the representative scatter values ( $S_{\text{for}}$  or  $S_{\text{bac}}$ ), ( $S_{\text{l,for}}$  or  $S_{\text{l,bac}}$ ) and ( $S_{\text{u,for}}$  or  $S_{\text{u,bac}}$ ).

**NOTE** The calculated standard deviation,  $\sigma_s$ , 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.2 Data reduction example

In Figure C.1, an arbitrarily selected scatter measurement scan is shown for a multilayer coating of  $\text{TiO}_2/\text{SiO}_2$  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.

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 per cent, the standard deviation,  $\sigma_s$ , and, consequently, the parameters ( $S_{\text{l,for}}$  or  $S_{\text{l,bac}}$ ) and ( $S_{\text{u,for}}$  or  $S_{\text{u,bac}}$ ) are drastically affected by a variation of the quantity  $dS$ .

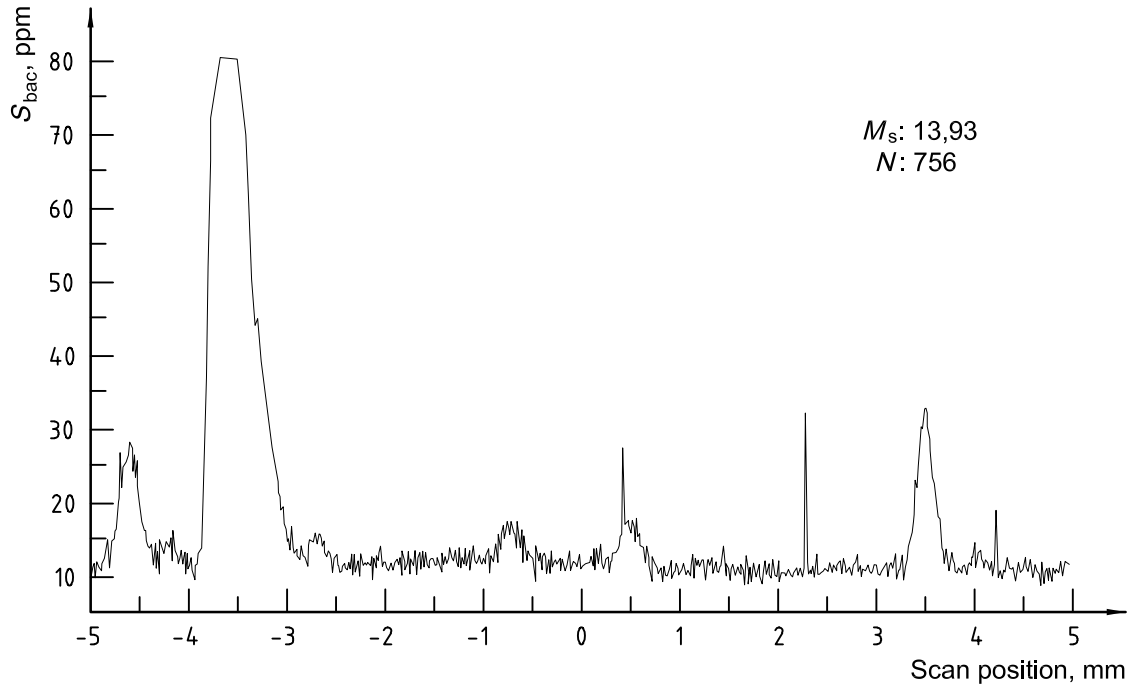
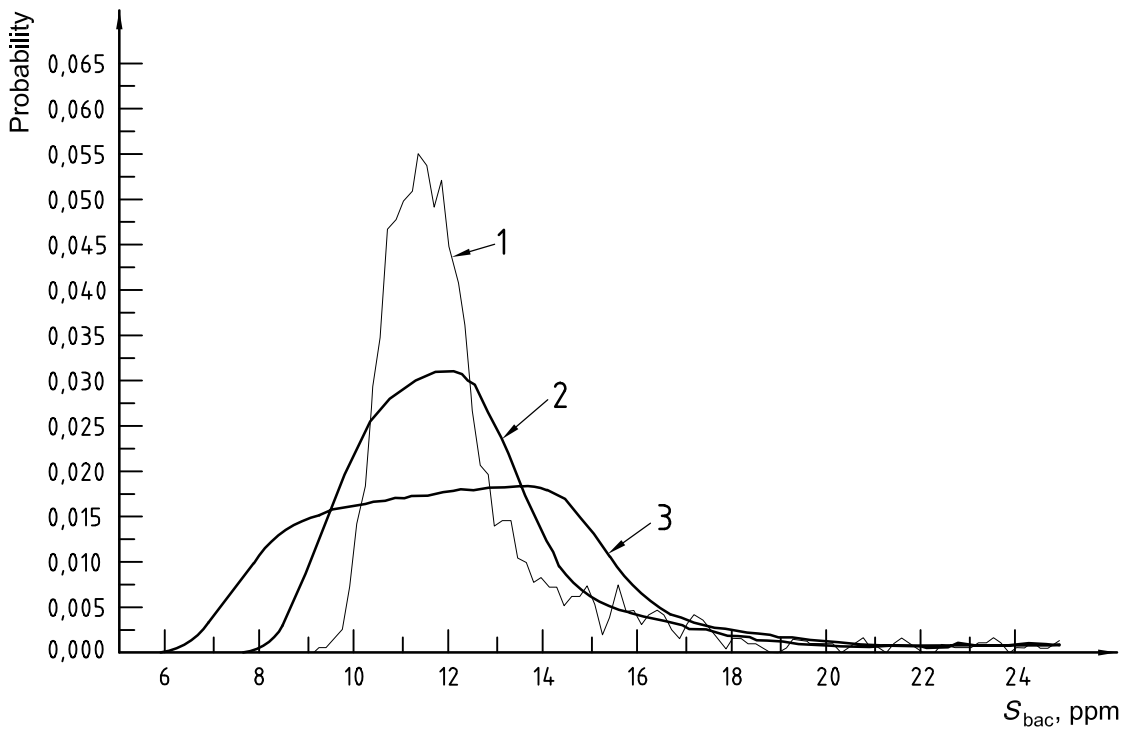


Figure C.1 — Total backward scattering values as a function of the scanning position



**Key**

1	$dS = 0,4$	$M_S = 11,37$	$\sigma_S = 0,91$ ppm
2	$dS = 4,0$	$M_S = 11,59$	$\sigma_S = 1,72$ ppm
3	$dS = 7,7$	$M_S = 11,84$	$\sigma_S = 2,97$ ppm

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

Figure C.2 — Distribution diagrams of the measured scatter values for selected values of the quantity  $dS$

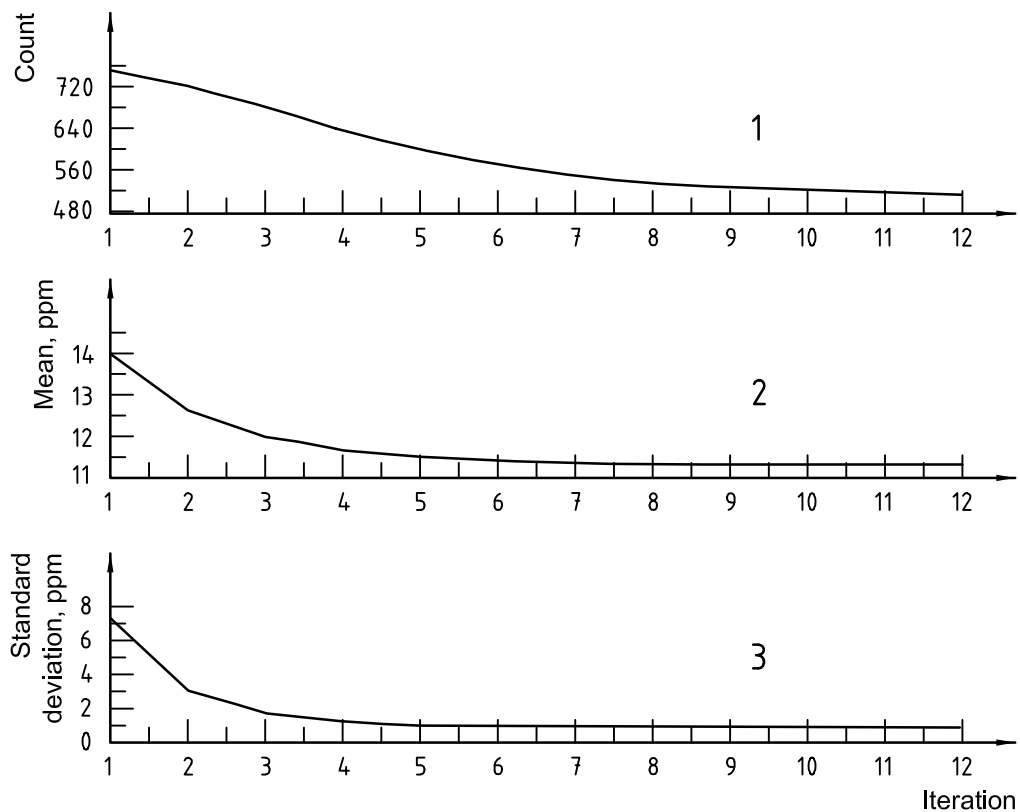
The development of the specific parameters  $M_S$ ,  $\sigma_S$  and the number of selected data points during the data reduction algorithm, is depicted in Figure C.3. After the steps 1 to 3 have been executed several times, the parameters stabilize to saturation values, which are characteristic of the intrinsic scatter level of the sample.

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

$$S_{bac} = 11,36 \text{ ppm}$$

$$S_{l,bac} = 10,72 \text{ ppm}$$

$$S_{u,bac} = 11,68 \text{ ppm}$$

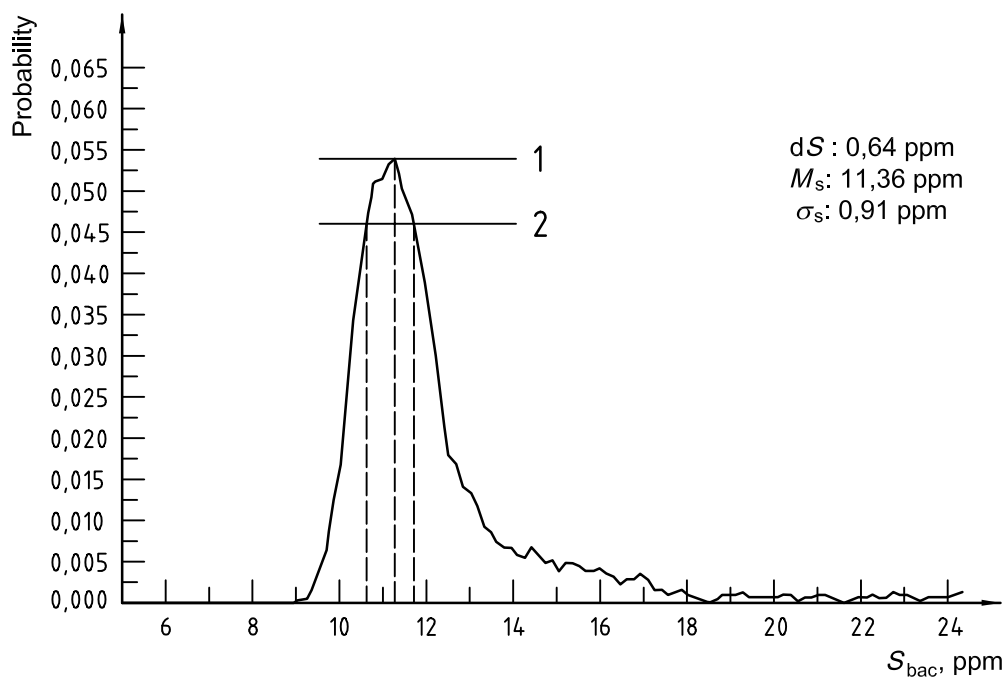


**Key**

- 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 parameters are depicted as a function of the iterative step number.

**Figure C.3 — Development of the specific parameters  $M_S$ ,  $\sigma_S$  and the number of selected data points during the data reduction algorithm**



**Key**

- 1 11,36 ppm
- 2 10,72 and 11,68 ppm

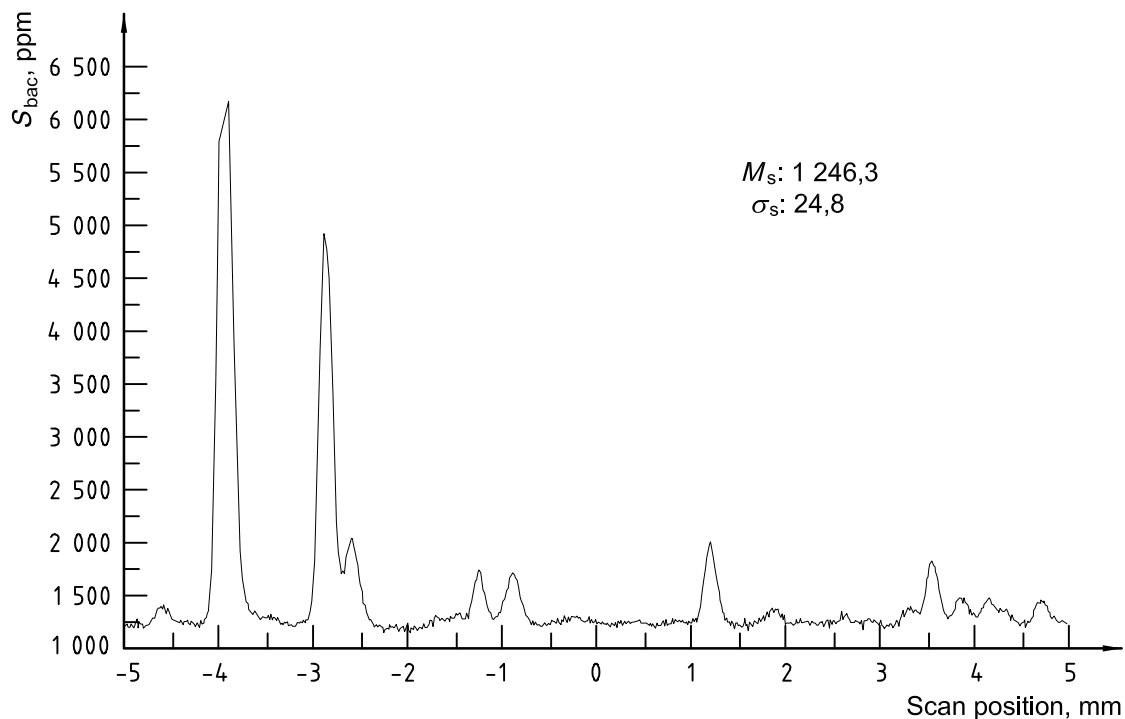
**Figure C.4 — Distribution diagram of the measured scatter values for the optimized value of the quantity,  $dS$**

## Annex D (informative)

### Example for selection of spacing

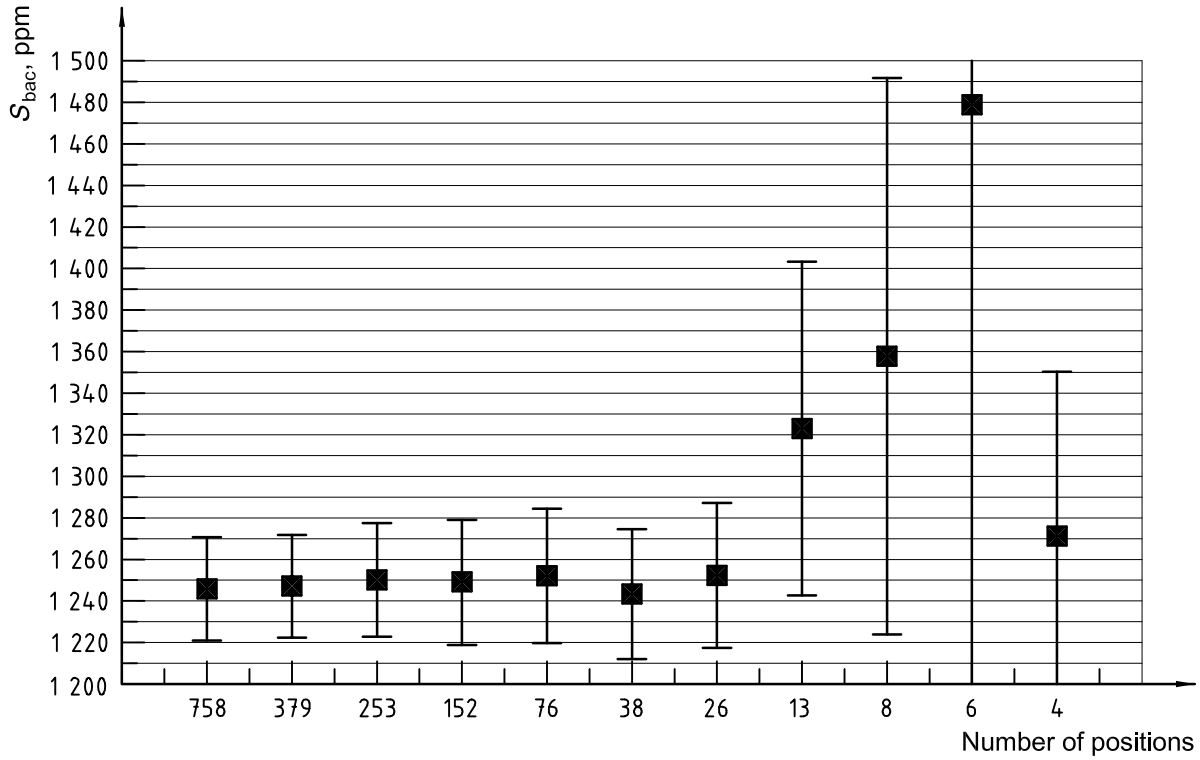
#### D.1 Scanning of the specimen

For scatter measurement systems with a scanning device, the selection of the spacing between adjacent measurement positions is dependent on the beam diameter, on the surface topography of the specimen and on the scanning length. As an example, the behaviour of the scatter value,  $S_{\text{bac}}$ , and the deviation of the scatter values is investigated for a typical optical surface scan depicted in Figure D.1. The results are plotted in Figure D.2 for different numbers of equidistant data points within a scan with constant length. For small spacing values, i.e. high numbers of data points in a single surface scan, the variation of the scatter value,  $S_{\text{bac}}$ , is significantly below its standard deviation. If the number of data points is reduced, the statistical distribution of surface defects with high scatter values contributes significantly to the variation of the scatter values,  $S_{\text{bac}}$ . For this example, the number of data points for a single scan should be higher than 30, in order to suppress the influence of localized contamination and scatter defects on the scatter value,  $S_{\text{bac}}$ .



NOTE The sample consists of an optically polished filter glass (RG 1 000) which is coated with a design (HL)<sup>6</sup>H of TiO<sub>2</sub>/SiO<sub>2</sub>.

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



NOTE 1 The data points are equally spaced and were chosen from the example in annex C.

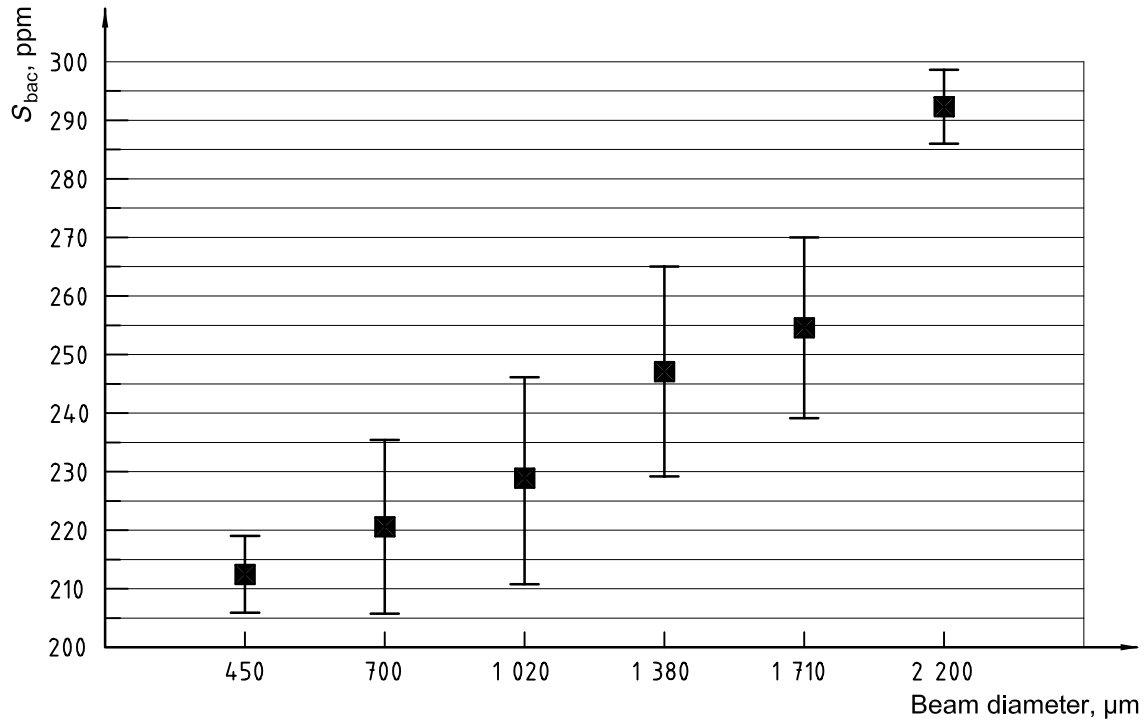
NOTE 2 Data reduction was performed as described in annex C.

**Figure D.2 — Scatter value,  $S_{bac}$ , as a function of the number of data points taken in a 10 mm long surface scan**

If the specific properties of the surface under test are not known at the beginning of the test, it is recommended to select the highest number of data points possible for the measurement set-up. Also, a fraction of the surface area, which is representative for the specific statistical surface properties, should be examined. The spacing of the measurement positions should be chosen significantly smaller than the beam diameter in order to identify localized scattering defects and to reduce influences by the noise of the measurement system and the environment.

## D.2 Influence of the beam diameter

Occasionally, an increase of the scatter value,  $S_{bac}$ , with the beam diameter of the scatter measurement device is observed for coated and uncoated optical surfaces (see, for example, Figure D.3). This behaviour may be attributed to contributions by localized scattering defects. If the mean distance between these surface features is of the same order of magnitude as the beam diameter, their contribution is no longer separable from the intrinsic fraction of the scattered light, resulting in an increased base level of scattering,  $S_{bac}$ . For typical optical surfaces with a high reflection coating that is very clean, a significant increase in the scatter value,  $S_{bac}$ , may be observed for beams larger than 1 mm in diameter.



NOTE 1 The scatter values were measured with the apparatus described in annex B with an increased exit port diameter of 16 mm.

NOTE 2 The values  $S_{\text{bac}}$  were evaluated according to the procedure described in annex C.

**Figure D.3 — Scatter,  $S_{\text{bac}}$ , as a function of the beam diameter for a high reflecting mirror coated with a standard dielectric stack (HL)<sup>15</sup>H of  $\text{Ta}_2\text{O}_5/\text{SiO}_2$**

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1) To be published. (Revision of ISO 12005:1999)



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