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

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

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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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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

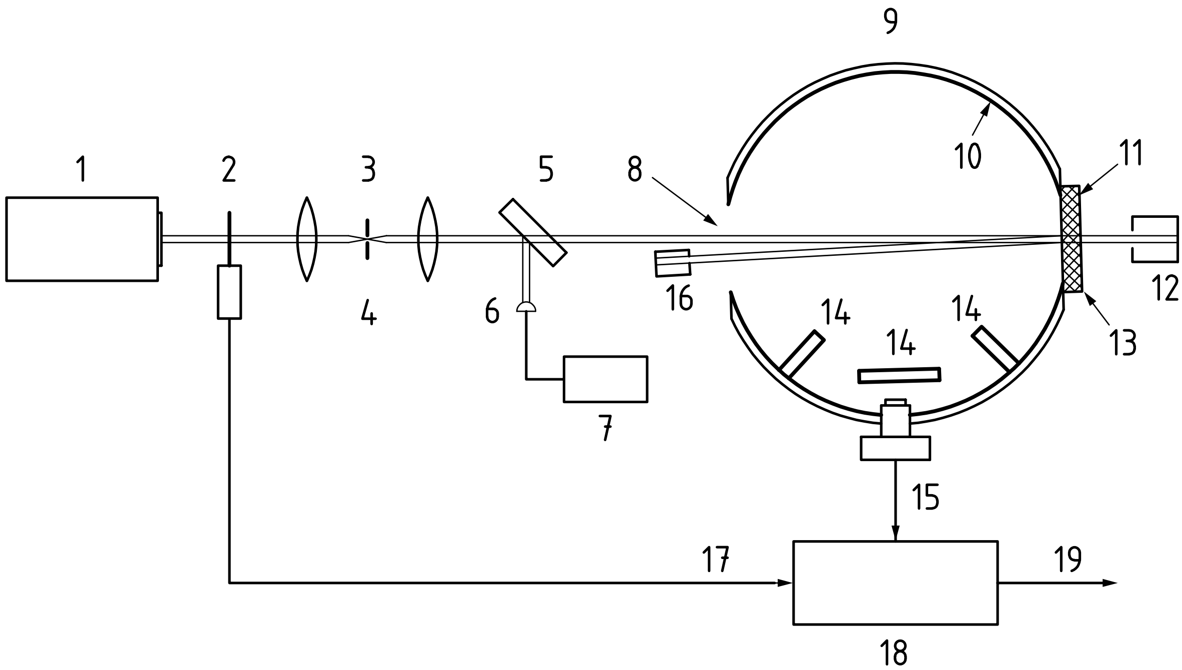
4.1 Principle

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

