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Optics and photonics — Measurement of reflectance of plane surfaces and transmittance of plane parallel elements

Optique et photonique — Mesurage du facteur de réflexion des surfaces planes et du facteur de transmission des éléments à plan parallèle

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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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.

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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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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 1, *Fundamental standards*.

This second edition cancels and replaces the first edition ISO 15368:2001 which has been technically revised. The main changes compared to the previous edition are as follows:

— Throughout the document, descriptions of the use of Fourier transform spectrometer instruments have been expanded and added where appropriate to an equivalent level as those of monochromator instruments.

— Throughout the document, the term “light” has been replaced with “optical radiation” to reflect that this standard’s spectral range extends beyond the visible.

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

Measurements of reflectance and transmittance using spectrophotometers are the most fundamental methods for the characterization of optical components. Since the spectrophotometric methods are basic and normal, they are extensively used and provide measurement data over a wide range of wavelengths.

This document describes the measurement of reflectance and transmittance using spectrophotometers, which provides data with high reproducibility and repeatability.

Optics and photonics — Measurement of reflectance of plane surfaces and transmittance of plane parallel elements

1 Scope

This document provides rules for the measurement of the spectral reflectance of plane surfaces and the spectral transmittance of plane parallel elements using spectrophotometers.

This document only applies to measurements of the regular transmittance and the regular reflectance; it does not apply to those of the diffuse transmittance and the diffuse reflectance.

This document is applicable to test samples, which are coated or uncoated optical components without optical power.

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 9211-1, *Optics and photonics — Optical coatings — Part 1: Vocabulary*

ISO 10110-8, *Optics and photonics — Preparation of drawings for optical elements and systems — Part 8: Surface texture*

ISO 80000-7, *Quantities and units — Part 7: Light and radiation*

ISO/IEC Guide 98-3, *Uncertainty of measurement — Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*

IEC 60050-845, *International Electrotechnical Vocabulary — Chapter 845: Lighting*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 9211-1, ISO 80000-7, IEC 60050-845 and ISO/IEC Guide 98-3, and the following apply.

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

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

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

3.1
transmittance
<for incident radiation of a given spectral composition, polarization and geometrical distribution> ratio of the transmitted radiant or luminous flux to the incident flux in the given conditions

[SOURCE: IEC 60050-845:1987, 845-04-59]

3.2
regular transmittance
ratio of the regularly transmitted part of the (whole) transmitted flux, to the incident flux

[SOURCE: IEC 60050-845:1987, 845-04-61]

3.3
diffuse transmittance
ratio of the diffusely transmitted part of the (whole) transmitted flux, to the incident flux

Note 1 to entry: $\tau = \tau_r + \tau_d$ (see also Clause 4).

Note 2 to entry: The results of the measurements of τ_r and τ_d depend on the instruments and the measuring techniques used.

[SOURCE: IEC 60050-845:1987, 845-04-63]

3.4
internal transmittance
ratio of the radiant or luminous flux reaching the internal exit surface of the layer to the flux that enters into the layer after crossing the entry surface

3.5
reflectance
<for incident radiation of a given spectral composition, polarization and geometrical distribution> ratio of the reflected radiant or luminous flux to the incident flux under the given conditions

[SOURCE: IEC 60050-845:1987, 845-04-58]

3.6
regular reflectance
ratio of the regularly reflected part of the (whole) reflected flux, to the incident flux

[SOURCE: IEC 60050-845:1987, 845-04-60]

3.7
diffuse reflectance
ratio of the diffusely reflected part of the (whole) reflected flux, to the incident flux

Note 1 to entry: $\rho = \rho_r + \rho_d$ (see also Clause 4).

Note 2 to entry: The results of the measurements of ρ_r and ρ_d depend on the instruments and the measuring techniques used.

[SOURCE: IEC 60050-845:1987, 845-04-62]

3.8
relative reflectance
ratio of the reflected flux from a sample to that from a reference

4 Symbols and units

For the purposes of this document, the following symbols and units apply.

λ	wavelength, expressed in nanometres
p, s	state of polarization
τ	transmittance
τ_r	regular transmittance

τ_d diffuse transmittance

τ_i internal transmittance

ρ reflectance

ρ_r regular reflectance

ρ_d diffuse reflectance

$\rho_{r,rel}$ relative regular reflectance

NOTE Wherever the Greek letters, ρ and τ, are mistakable, T and R can be used.

5 Test sample

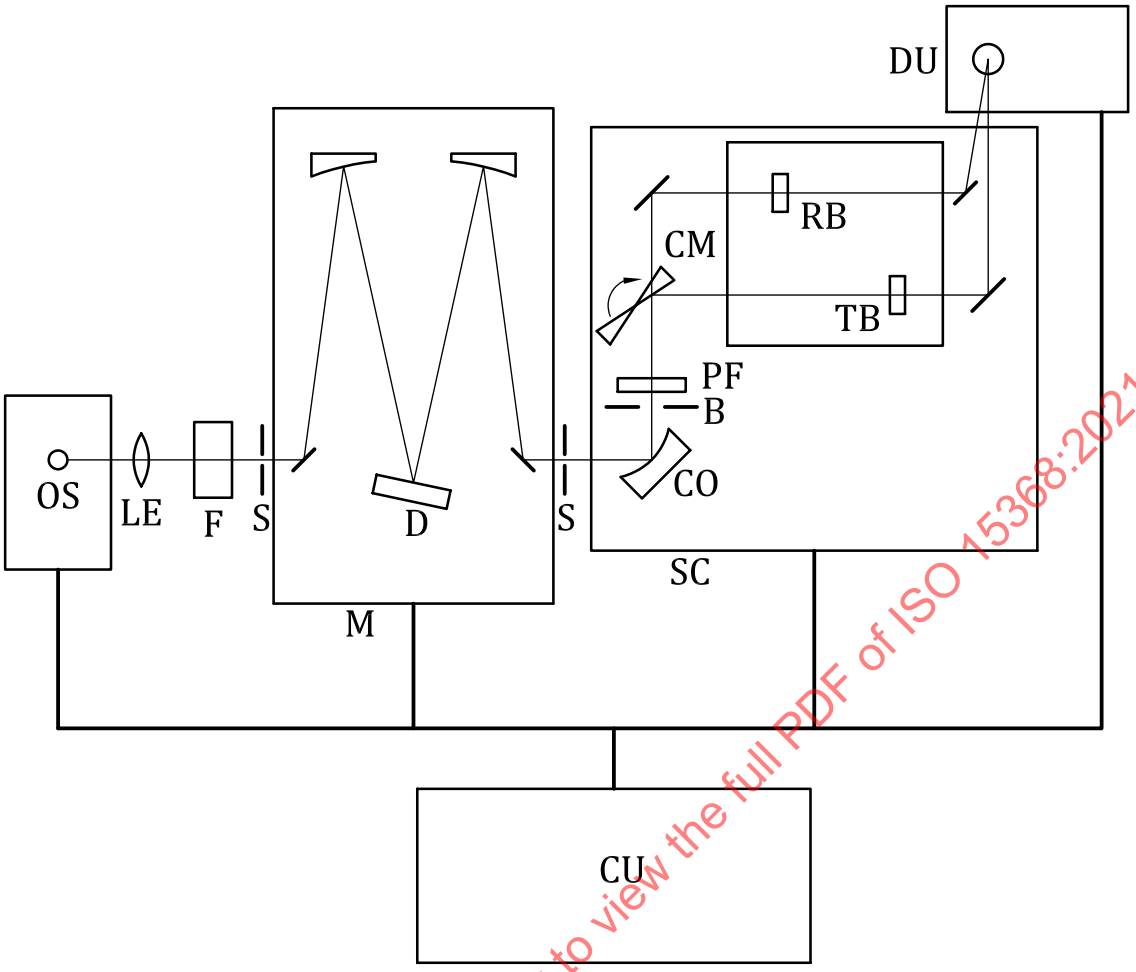
The storage, cleaning and preparation of a test sample shall be carried out in accordance with the instructions of the manufacturer on the test sample for normal use.

The wavelength, angle of incidence and state of polarization shall correspond to those specified by the manufacturer for the use of the test sample.

6 Measuring apparatus

For the measurements specified in this document, a spectrophotometer is required. Figure 1 shows an example of a double beam, dispersive type spectrophotometer. Figure 2 shows an example of a single beam, interferometer type Fourier-transform spectrophotometer (FTS). Both types consist of an optical radiation source, a spectral unit, a sample compartment, a detector unit and a control unit.

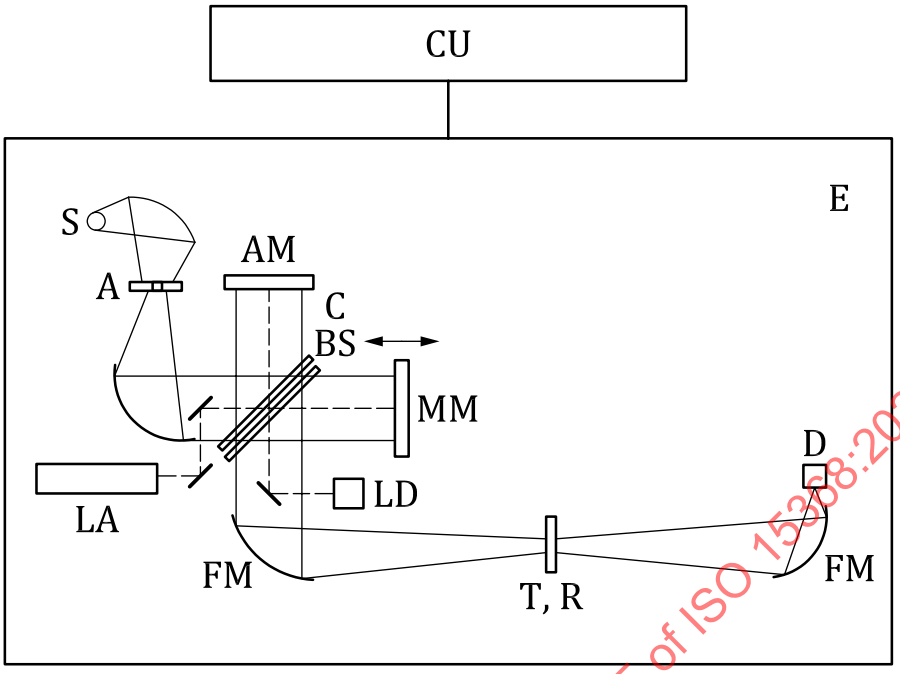
Details of the apparatus are described in Annex A.



Key

OS	optical radiation source	B	baffle
LE	lens	PF	polarization filter
F	filter box	CM	chopper mirror
S	slit	TB	test beam and test sample
D	dispersive element	RB	reference beam and reference sample
M	monochromator	DU	detector unit
SC	sample compartment	CU	control unit
CO	collecting optics		

Figure 1 — Typical arrangement of a dispersive spectrophotometer



Key

E	enclosure	MM	moving mirror
S	source	T, R	sample or reference for transmittance and reflectance
A	aperture	LA	laser
BS	beam splitter	LD	laser detector
C	compensator plate	CU	control unit
AM	alignment mirror	D	detector unit
FM	focusing mirror		

Figure 2 — Typical arrangement of a Fourier-transform spectrophotometer

7 Test conditions

7.1 Dispersive type spectrophotometer

7.1.1 General

The optical radiation source, the divergence of the beam, the beam diameter on the sample, the wavelength, spectral resolution, the stepping interval, the incident angle, the detector and any required numerical correction shall be selected and documented.

7.1.2 Optical radiation source

The temporal variation of the intensity of the optical radiation source shall be measured and documented. The state of polarization (*p* or *s*) of the beam shall be selected and documented.

The state of polarization of the radiation reaching the detector can be affected by reflection on components in the reference/sample paths. It is suggested to tilt a transmitting sample by equal amounts in orthogonal directions to check for polarization effects. The beam diameter on the sample shall be larger than 1 mm. On the surface of the sample, the beam profile shall be smooth so that the local peak power density does not exceed the average power density by a factor of greater than or equal to two. The beam diameter and f–number or numerical aperture (see also 9.11) shall be documented.

7.1.3 Monochromator

The type of dispersive element and its characteristics shall be documented.

Optics for blocking out higher order diffracted optical radiation shall be documented.

The spectral range and spectral resolution shall be selected in order to satisfy the specification of the measurement and documented.

The type of spectrophotometer (single or double beam) shall be documented.

7.1.4 Detection system

An appropriate detector for the measured spectral region shall be selected and documented. A lock-in detection technique is frequently used and an optical radiation chopper or a chopper mirror is installed in the beam to modulate the output signal. The detection system shall have a dynamic range greater than 10^4 and a deviation from linearity less than 10^{-2}. Photometric linearity shall be calibrated by a double aperture method that uses double apertures and neutral density filters[4].

When an integrating sphere or a diffuser is used, it shall be documented.

7.1.5 Numerical correction

Numerical correction can include spectral correction, averaging, smoothing, calibration of the photometric linearity and other factors.

A spectral correction can be applied using an appropriate wavelength standard (see 9.2). Random noise can be reduced by averaging or smoothing. Averaging can be performed by repeating measurements or increasing sampling time. Smoothing can be achieved by averaging data over a finite spectral bandwidth after measurement, although it will reduce the spectral resolution. The sampling time and smoothing factors shall be documented.

For details on the calibration of photometric linearity, see 7.1.4.

Calibration of the spectrophotometer can be performed by measuring the transmittance of a reference sample (standard) using the method provided in 8.2.1. A reference sample for transmittance from the ultraviolet to the near infrared region shall be a parallel plate of fused silica with a P2 grade surface as specified in ISO 10110-8. Refractive index data for undoped float-zone Si over the spectral range from 1,2 µm to 5,5 µm, and high purity Ge from 1,7 µm to 23 µm can be used as reference standards. The expanded uncertainty of the measurement of the transmittance of the reference sample shall be between 0,02 % and 1 %. This shall include repeatability and photometric noise, for $k = 2$ (95 % confidence). Other standard reference materials, which are checked at an accredited laboratory may be used.

7.2 Fourier-transform type spectrophotometer

7.2.1 General

The optical radiation source, sample incident beam geometry (central angle of incidence, f-number or half-angle and spot size on the sample), the wavenumber (or wavelength) range, spectral resolution, sampling interval, detector and numerical correction shall be selected and documented.

7.2.2 Optical radiation source

The type of optical radiation source shall be documented. The temporal variation of the intensity of the optical radiation source, indicated by the interferogram signal level, shall be measured and documented. If the state of polarization (*p* or *s*) of the beam is important, then the polarization shall be controlled and documented.

NOTE The state of polarization of the radiation reaching the detector can be affected by reflection on components in the beam path.

The aperture size shall be selected to be consistent with the spectral resolution setting determined by the optical beam path difference in the interferometer.

The sample size shall be larger than the beam diameter.

7.2.3 Interferometer

The type of beam splitter/compensator plate and its characteristics shall be documented.

The type of interferometer scanning mode (continuous scan or step scan), and interferogram scanning range (or corresponding spectral resolution) and sampling interval (or corresponding spectral range), shall be documented. The spectral range and spectral resolution shall be selected in order to satisfy the specification of the measurement.

7.2.4 Detection system

An appropriate detector for the measured spectral region shall be selected and documented. The detection system shall have a dynamic range greater than 10^3 and a deviation from linearity less than 10^{-2}. In contrast to a dispersive spectrophotometer, linearity cannot be calibrated by a double aperture method, and another method such as use of attenuating filters or multiple standards shall be selected.

When an integrating sphere or a diffuser is used, this shall be documented.

As part of the data acquisition, directly measured interferograms are processed through software to obtain resulting spectra. For details see also ISO 19702 and Reference [6].

7.2.5 Numerical correction

Numerical correction can include spectral correction, averaging, smoothing, calibration of the photometric linearity and other factors.

The manner and parameters of phase correction (self- or reference-phase, and number of interferogram data points), interferogram apodization and zero-filling, and spectral shift corrections shall be documented.

A spectral correction can be applied using an appropriate wavelength standard (see 9.3). The typical correction is directly proportional to wavenumber. Random noise can be reduced by averaging or smoothing. Averaging can be performed by repeating measurements or increasing sampling time, which is determined by a combination of the mirror scanning speed, spectral resolution, and the number of scans per measurement. Smoothing can be performed by averaging data over a finite spectral bandwidth after measurement, although it will reduce the effective spectral resolution. The sampling time and smoothing factors shall be documented.

Calibration of the spectrophotometer can be performed by measuring the transmittance of a reference sample (standard) using the method given in 8.2.1. Refractive index data for undoped float-zone Si over the spectral range from 1,2 µm to 5,5 µm, and high purity Ge from 1,7 µm to 23 µm can be used as reference standards. The expanded uncertainty, including repeatability, of the transmittance of these reference samples is from 0,3 % to 1 % including photometric noise. For longer wavelengths, few standards are available, other than reference samples, which have been calibrated at an accredited laboratory, such as a National Metrology Institute.

8 Test procedure

8.1 Measurement of reflectance

8.1.1 General

One of two types of measurement of reflectance, a direct method, or a relative method, shall be chosen. When the relative method is used, either the regular reflectance or the relative regular reflectance is obtained, depending on knowledge of the reference reflectance.

The incident angle shall be selected according to the manufacturer's instructions. Reflectance at normal incidence cannot usually be measured and an incident angle between 5° and 15° instead of 0°, which shall be documented, is typically used. In the case of an incident angle other than 0°, the reflectance depends on the state of polarization of the incident optical radiation, so that in the case of an angle larger than 10°, the state (*p* or *s*) shall also be selected and documented.

8.1.2 Direct measurement of regular reflectance

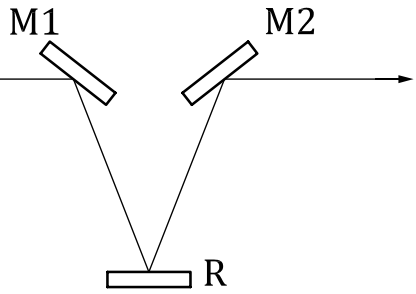
Figure 3 shows three methods used for the direct measurement of reflectance. In Figure 3 a) the reflected flux Φ_1 without a sample is measured, and then in Figure 3 b) and c), the reflected flux Φ_2 with the sample is measured after changing the optical arrangement as shown. In Figure 3 d) the reflected flux Φ_2 with the sample is measured in the configuration shown on the left side of the figure, followed by the incident flux measurement in the configuration shown on the right side of the figure. The regular reflectance of the sample is given by Formula (3):

$$\rho_r = \frac{\Phi_2}{\Phi_1} \tag{3}$$

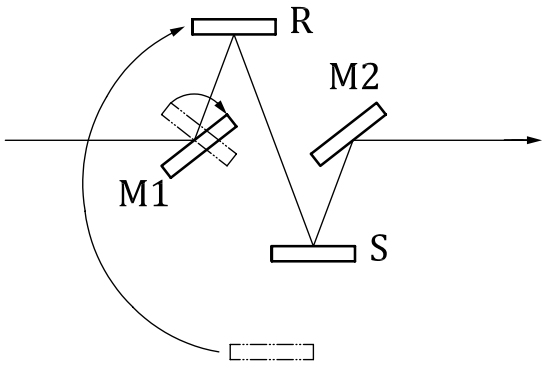
in the case of the arrangements shown in Figure 3 b). The regular reflectance is given by Formula (4):

$$\rho_r = \sqrt{\frac{\Phi_2}{\Phi_1}} \tag{4}$$

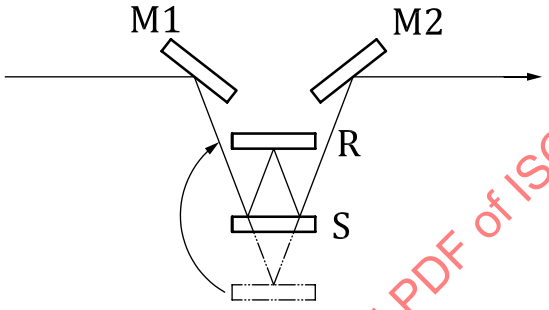
in the case of the arrangements shown in Figure 3 c), irrespective of the magnitudes of the reflectance of the reference mirror and other optics.



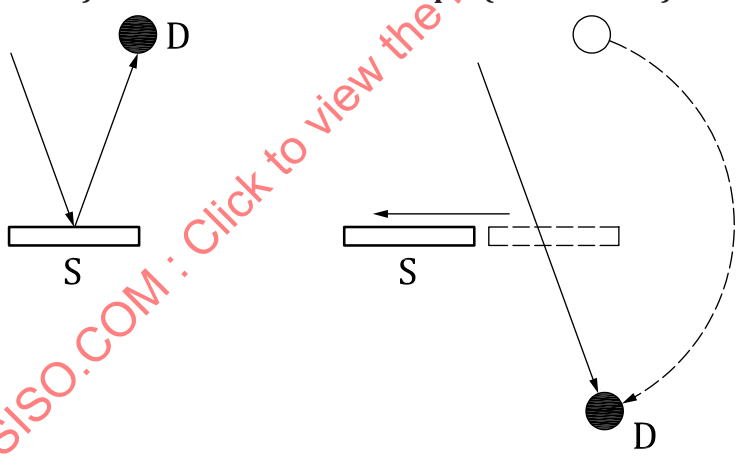
a) Reference measurement



b) Measurement of the sample (V-N method)



c) Measurement of the sample (V-W method)



d) Measurement of the sample (goniometer method)

Key

M1	mirror 1	S	sample
M2	mirror 2	D	detector
R	reference mirror		

Figure 3 — Direct measurement of reflectance

8.1.3 Relative measurement of regular reflectance

The relative measurement is easier than the direct measurement. An example of a reference sample for the reflectance is an aluminium or gold mirror or a fused silica plate with a wedge angle, polished smoothly and kept clean. Successive measurements of the reflected flux of the reference sample Φ_1

and that of a sample Φ_2, where the sample is substituted for the reference sample are made using the arrangement of Figure 3 a). Then the regular reflectance of the sample is given by Formula (5):

$$\rho_r = \frac{\Phi_2}{\Phi_1} \times \rho_{ref} \tag{5}$$

where ρ_{ref} is the regular reflectance of the reference sample.

The value ρ_{ref} is calibrated separately by a direct method given in 8.1.2 or obtained as described below. For a low reflectance sample such as an anti-reflection coated or uncoated glass plate, the relative measurement is recommended. In such a case, a low reflectance material calibrated by an accredited laboratory or a fused silica plate, which is sufficiently wedged to avoid multiple reflections from being measured, is used as the reference sample for the region from the ultraviolet to the near infrared. The reflectance of the fused silica plate shall be numerically calculated from its refractive index. The refractive index of the fused silica is given in Annex B.

Other standard reference materials for which reliable refractive index data is available may be used and shall be used for wavelengths longer than 3 µm (wavenumbers less than 3 300 cm^{-1}), when no material calibrated by an accredited laboratory is available.

In the case of the measurement of the first-surface regular reflectance of a transparent glass plate, for example, reflection from the second-surface shall be eliminated. Reflection from the second surface can cause a significant error, unless eliminated. One method of eliminating this reflection is to make the sample wedge-shaped. In this case, secondary reflections should be checked to ensure that they do not contribute to optical radiation reaching the detector; another method is to grind the second surface and coat it with absorbing paint. For the infrared, grinding the back surface could be insufficient to scatter optical radiation at long wavelengths.

In the case of the measurement of the regular reflectance of a second surface mirror, the reflection from the first surface is probably not completely eliminated. In this case, a comment shall be added.

8.1.4 Measurement of relative reflectance

When a comparison of samples to a reference is of interest, as in the case of monitoring a process, but the reflectance of the reference sample is unknown, the relative reflectance can be obtained through a ratio measurement identical to that described in 8.1.3, using the arrangement of Figure 3 a). Then the relative regular reflectance of the sample is given by Formula (6):

$$\rho_{r,rel} = \frac{\Phi_2}{\Phi_1} \tag{6}$$

8.2 Measurement of transmittance

8.2.1 Regular transmittance

For the measurement of regular transmittance, the flux with (w) and without (wo) a sample is measured. The ratio of the regularly transmitted flux with the sample to that without the sample gives the regular transmittance as given by Formula (7):

$$\tau_r = \frac{\Phi_w}{\Phi_{wo}} \tag{7}$$

The regular transmittance is affected by the reflection from the boundary surfaces. Even a sample without any absorption or scattering has a transmittance less than 100 % (around 92 %, for a glass plate).

8.2.2 Internal transmittance of an optical plate

In the case of an optical plate, the internal transmittance is sometimes important. For the measurement of the internal transmittance, the reflections from the first and the second surfaces shall be compensated. Two plates with different thicknesses, e.g. 2 mm and 12 mm, are prepared and their regular transmittance τ_{r2} and τ_{r12} is measured. The internal transmittance τ_i for a sample of 1 cm thickness is given by Formula (8):

$$\tau_i = \frac{\tau_{r12}}{\tau_{r2}} \tag{8}$$

The above formula is valid when internal multiple reflections are ignored. For a sample with very low transmittance, that is, of high absorptance, internal multiple reflections can be ignored because multiply reflected optical radiation is almost completely absorbed. For the sample of high transmittance, internal multiple reflections can also be ignored because the intensities of the multiply reflected and transmitted optical radiation are almost the same for both τ_{r2} and τ_{r12}.

However, for the medium transmittance sample, the error caused by internal multiple reflections in the internal transmittance calculated by the formula reaches 0,1 %, which can be reduced by numerical correction if the reflectance and transmittance of boundary surfaces are known.

9 Common sources of error

9.1 General

The primary types of error of the spectrophotometer are wavelength (or wavenumber) error, wavelength reproducibility, spectral resolution, fluctuation of the incident flux (optical radiation source), parallelism of the sample, stray optical radiation, linearity of the detection system, misalignment of the sample, inter-reflections, baseline reproducibility and beam divergence. They are briefly described in 9.2 to 9.11; however, it is not feasible to indicate their combination for the evaluation of measurement uncertainty in a general case.

Typical values of the overall photometric expanded uncertainty of the spectrophotometer are 0,3 % in the ultraviolet and visible region (190 nm to 780 nm), and 1,3 % in the infrared region (0,78 µm to 50 µm or 200 cm^{-1} to 12 800 cm^{-1}). These values assume careful attention to the potential instrumental sources of error and application of corrections where appropriate and feasible.

9.2 Monochromator wavelength uncertainty, reproducibility and spectral resolution

Wavelength expanded uncertainty, reproducibility and spectral resolution of the typical monochromator are given in Table 1.

Table 1 — Typical values for the expanded uncertainty, reproducibility and spectral resolution in the different spectral ranges

Wavelength range	Expanded uncertainty	Reproducibility	Spectral resolution
190 nm to 780 nm	0,2 nm	0,1 nm	0,1 nm
780 nm to 2 500 nm	1 nm	0,5 nm	0,1 nm
2 500 nm to 25 000 nm (2,5 µm to 25 µm)	1 cm^{-1} to 5 cm^{-1}	0,5 cm^{-1} to 5 cm^{-1}	0,1 cm^{-1} to 8 cm^{-1}

Wavelength uncertainty, reproducibility and spectral resolution shall be documented.

The wavelength scale can be calibrated by several wavelength standards such as spectral lamps and a holmium and a didymium glass plate in the visible (380 nm to 780 nm) and near infrared (0,78 µm to

3 µm) regions. In the mid infrared region (3 µm to 50 µm), absorption spectra of polystyrene, indene, toluene, trichlorobenzene, etc. and rotation-vibration spectra of CO_2, CO, HCl, H_2O, etc. are used.

NOTE For further information regarding the spectral ranges see ISO 20473.

9.3 Interferometer wavenumber uncertainty, reproducibility and spectral resolution

The wavenumber expanded uncertainty of FTS instruments can vary between 0,01 cm^{-1} and 2 cm^{-1} at 10 000 cm^{-1}, depending on instrument design and alignment of the infrared source optical radiation with the laser reference optical radiation (which is used to set the wavenumber scale). For FTS instruments, the wavenumber uncertainty is typically proportional to the wavenumber value. The wavenumber reproducibility error is typically insignificant. The spectral resolution of an FTS instrument is variable across a range from a lower limit of between 0,01 cm^{-1} and 4 cm^{-1} to an upper limit of typically 32 cm^{-1}. The spectral resolution is independent of the wavenumber value.

Numerical correction of FTS interferograms and spectra (phase correction, apodization, zero-filling, and spectral shift corrections; See 7.2.5 and Reference [6]) should be consistent among different manufacturers or organizations for reproducibility of measurements.

The wavenumber scale can be calibrated by several wavelength standards such as spectral lamps and a holmium and a didymium glass plate in the visible (380 nm to 780 nm) and near infrared (0,78 µm to 3 µm) regions, as well as calibrated polystyrene in the mid infrared region (3 µm to 50 µm). In the mid infrared region, for instruments with sufficient resolution to distinguish multiple bands, absorption spectra of polystyrene, indene, toluene, trichlorobenzene, etc. and rotation-vibration spectra of CO_2, CO, H_2O, HCl, etc. are used.

Wavenumber uncertainty, reproducibility (if significant) and spectral resolution shall be documented.

9.4 Fluctuation of the incident flux

Typical fluctuation of the optical radiation source depends on the stability of the electric source and the environmental condition. In the case of the single beam spectrophotometer, the fluctuation of the incident flux shall be monitored. In the case of the double beam spectrophotometer, the fluctuation can be monitored and compensated by the reference beam. Another monitoring system can also be used. Repeated measurements can be used to characterize the effects of fluctuations.

9.5 Parallelism of the sample

In the case of the measurement of regular reflectance, the reflectance of a single surface is often of primary interest. In such a case, reflection from the other surface shall be eliminated, as for example, for the measurement of an anti-reflection coating or a semi-transparent mirror. In contrast, when the total reflectance is of interest, collection of the additional reflections shall be included. In such a case, an averaging sphere can prove useful to obtain complete collection of all reflections.

In the case of the measurement of transmittance, the measured value can depend on the wedge angle between the first and second surfaces. When the sample surfaces are parallel, multiple-reflected beams from both surfaces are collected by the detector. The error caused by these beams can be numerically corrected if the reflectance and transmittance of both surfaces are known. On the other hand, when an appropriate wedge angle is used, any multiple-reflected beam from both surfaces is not collected by the detector. As for reflectance, an averaging sphere can prove useful.

When a wedge angle is used, it shall be documented.

NOTE See also ISO 9211-2:2010, 8.1.2, 8.2.2 and Annex A.

9.6 Monochromator stray optical radiation

There are two types of stray optical radiation: heterochromatic and homochromatic. Heterochromatic stray optical radiation originates inside the monochromator. Homochromatic stray optical radiation originates from the reflection from the optics, the sample and the detector.

A good monochromator has low heterochromatic stray optical radiation, typically 0,000 1 % in the ultraviolet and visible regions, and 0,1 % in the near infrared region.

9.7 Linearity of the detection system

Calibration and testing of linearity of the detection system is described in 7.2.4. In FTS instruments, nonlinearity can result in false signals at wavenumbers that are multiples of the strongest signals within the measured spectra. Pyroelectric detectors are generally very linear but also temperature dependent, and benefit from temperature stabilization. Photo-conductive detectors including InSb and MCT detectors can exhibit significant non-linearity at high signal levels.

9.8 Inter-reflections

Inter-reflections between the multitude of components within the instruments can result in error and corresponding uncertainty.

For monochromator instruments, in the case of the measurement of transmittance, the reflected optical radiation from the sample causes stray optical radiation or a ghost. To reduce it, the sample shall be tilted slightly in relation to its nominal position (perpendicular to the beam).

For FTS instruments, where the solid angles of focused beams at the source, aperture, sample and detector are typically greater than in monochromator systems, small tilts of the sample are typically insufficient to completely remove inter-reflections. This is a consequence of the need to maximize the flux available for sufficient signal-to-noise in the infrared. Effects of inter-reflections include elevated transmittance and reflectance values, unexpected interference structure in spectra, and false structure in unexpected locations in spectra (due to double modulation by the interferometer). Approaches to reduce the inter-reflections include the use of half-beam blocks or half-field stops, an example of which is described in A.3.5.

9.9 Misalignment of the sample

Influence of misalignment of the sample depends on the characteristics of the detector. When the test beam is shifted or tilted by a misalignment of the sample, the angle or the position of the incident beam changes and the sensitivity of the detector can change. To eliminate this error and to assure reproducibility of the measured value, an appropriate positioning device shall be used [see also A.2.4 and A.3.6].

Thick and/or high index samples can axially displace the focus on the detector causing an erroneous result. This is particularly the case where small detectors are used, as in some infrared spectrophotometers. The effect is exacerbated by extreme off-axis detection system optics that are used in certain instruments.

9.10 Monochromator baseline reproducibility

Reproducibility and noise level of the baseline (100 % transmittance level) is between ±0,3 % and ±0,5 % in the visible and near infrared spectral regions.

9.11 Beam divergence

The typical beam divergence of monochromator instruments is less than 5°. For FTS instruments, within the interferometer, the beam divergence will vary with source aperture size, resulting in a limitation to the spectral resolution as well as causing a spectral shift, which can be corrected for. Therefore, the FTS

aperture size shall be documented. For FTS instruments, at the sample, the beam is typically focused to maximize the signal-to-noise ratio with f-numbers that can be as low as 3, equivalent to a maximum incident angle of 18°.

10 Test report

The test report shall include the following information:

a) Information on the test organization:

   1) Testing organization;

   2) Date of test;

   3) Examiner.

b) Information about the test sample:

   1) Manufacturer of the sample;

   2) Specifications of the manufacturer for storage and cleaning;

   3) Specifications of the manufacturer for normal use (spectral characteristics, wavelength, polarization, angle of the incident flux, purpose of use);

   4) Part identification code, date of production.

c) Information about the test:

   1) Test equipment [type of spectrometer (single or double beam, monochromator or Fourier-transform), optical radiation source, higher order suppression, beam splitter, detector, with or without integrating sphere or diffuser, detection system];

   2) Test conditions [incident angle, spectral range, stepping interval (monochromator), data interval (FTS) spectral resolution, polarization, beam diameter, beam divergence, edge angle of sample (if it is large)];

   3) Parameters of the detection system (scanning speed, sampling time or number of scans, numerical filtering, averaging and smoothing factors);

   4) Error budgets for wavelength and photometric uncertainty [wavelength reproducibility, photometric reproducibility, fluctuations of incident flux (optical radiation source), parallelism of the sample, stray optical radiation, detector linearity, baseline reproducibility (monochromator), alignment, uncertainty of the reference sample, etc.]

   5) Environmental conditions (temperature, degree of cleanness when a cleanroom is used, vacuum or purge gas).

d) Results:

   A graph and/or a table of spectral characteristics of the sample.

e) A reference to this document, i.e. ISO 15368:2021.

Annex A
(informative)

Spectrophotometers

A.1 General

This annex describes spectrophotometers used in the spectral range of 190 nm to 50 000 nm (0,19 µm to 50 µm; 52 600 cm^{-1} to 200 cm^{-1}).

A.2 Dispersive type spectrophotometer

Typical components of the spectrophotometer (see Figure 1) are as follows.

A.2.1 Optical radiation source

A D_2 (deuterium) lamp is used in the ultraviolet spectral region from 0,19 µm to 0,35 µm, a tungsten (halogen) lamp in the ultraviolet, visible and near infrared spectral regions from 0,25 µm to 3 µm and a Globar (SiC) or Nernst Glower (helically wound nichrome wire) in the infrared spectral region from 2,5 µm to 50 µm.

A.2.2 Monochromator

The monochromator is set between the optical radiation source and the sample compartment or between the sample compartment and the detector unit. The former arrangement has the advantage of reducing the radiant heat incident on the sample. The latter arrangement is used in the case of measurement of a fluorescent sample or the case using a linear array sensor. In the mid infrared region, the latter arrangement is also used for reducing heat radiation from the sample incident on the detector.

The dispersive element is a diffraction grating (0,19 µm to 50 µm), a glass prism (0,4 µm to 2 µm), a fused silica prism (0,19 µm to 3 µm) or a KBr prism (2,0 µm to 25 µm). For blocking the higher order optical radiation diffracted from a grating, an auxiliary prism or an absorption filter (a glass filter in the ultraviolet and visible region and an interference filter in the infrared region) is used.

For the measurement of high spectral resolution and/or low stray optical radiation, a double monochromator is recommended.

A.2.3 Sample compartment

The sample compartment has collecting optics, a polarization filter, a chopper (or a chopper mirror), test and reference beams, and baffles.

A.2.3.1 Collecting optics

Collecting optics are lenses or mirrors. Lenses often have the advantage of less aberration, whereas mirrors have the advantages of no chromatic aberration and less stray optical radiation.

A.2.3.2 Chopper

A chopper is used for lock-in detection. A chopper mirror can be used for both lock-in detection and switching of a test and a reference beam.

A.2.3.3 Polarization filter

A polarization filter is inserted when the reflectance at a large incidence angle (larger than 10°) is measured. In the spectral range between 0,22 µm and 2,3 µm, a polarizing prism made of birefringent crystal is available. In the spectral range between 0,28 µm and 2,0 µm, a polarization filter made of a sheet polarizer is convenient. Beyond 2,0 µm, wire grid polarizers are used, with spectral ranges depending on the grid spacing and substrate materials used, out to 50 µm.

A.2.3.4 Test and reference beam

The spectrophotometer is divided into two types; a single beam and a double beam (i.e. separate test and reference beams). The double beam type has the advantage of compensating fluctuations of the optical radiation source.

When a low transmittance or reflectance sample is measured, a neutral density filter can be inserted into the reference beam to balance the signal intensity.

A.2.3.5 Baffle

In order to shape and collimate the beam and to eliminate stray optical radiation, several baffles are usually used together with collecting optics. The baffle is usually placed on the plane conjugate to the exit slit or to the plane of the dispersive element of the monochromator and is usually focused on the sample or, if possible, on the detector.

A.2.4 Detector unit

A photomultiplier or a silicon photodiode is used in the ultraviolet and visible regions, a silicon photodiode, a germanium photodiode or an uncooled or cooled (reduced thermal noise) PbS cell in the wavelength region from 1 µm to 3,3 µm, and a thermocouple or vacuum thermopile in the wavelength region from 2,5 µm to 50 µm.

The sensitivity of most detectors, especially photomultipliers, depends on the angle, position and polarization of the incident beam. To reduce this dependence, an integrating sphere or a high transmittance or reflectance diffuser is installed in front of the detector. The magnitude of the signal is then largely reduced.

A.2.5 Control unit

Most photometers use lock-in detection, computer-controlled operation and numerical corrections. Lock-in detection greatly reduces photometric noise. Computer controlled operation can include scanning of the wavelength, controlling of the sensitivity of the detector, calculation of the ratio of the intensities of the test and the reference beam, calculation of the transmittance or reflectance and displaying of the data. Numerical correction can include spectral correction, averaging, calibration of photometric linearity and others.

A.3 Fourier-transform type spectrophotometer

A.3.1 General

In the spectral range between 2,5 µm and 50 µm (4 000 cm^{-1} and 200 cm^{-1}), an FTS is widely used, and often as well in the spectral range between 1 µm and 2,5 µm (10 000 cm^{-1} and 4 000 cm^{-1}). The detected signal is obtained as an interferogram, with the interferometer's optical path difference (cm) as the independent variable. Applying a Fourier transform to the interferogram results in a spectrum as a function of wavenumber (cm^{-1}).

Typical components of the spectrophotometer (Figure 2) are as follows. In addition to the brief details described below, ISO 19702 provides further details of Fourier-transform spectrophotometers and the related spectral calculations processes, including terminology.

