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Optics and optical instruments — Measurement of reflectance of plane surfaces and transmittance of plane parallel elements

Optique et instruments d'optique — Méthode de mesurage de la réflectance des surfaces planes et de la transmittance des éléments à plan parallèle

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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 15368 was prepared by Technical Committee ISO/TC 172, *Optics and optical instruments*, Subcommittee SC 1, *Fundamental standards*.

Annexes A and B of this International Standard are for information only.

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 further give measurement data for a wide range of wavelengths.

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

Optics and optical instruments — Measurement of reflectance of plane surfaces and transmittance of plane parallel elements

1 Scope

This International Standard gives rules for the measurement of the spectral reflectance of plane surfaces and spectral transmittance of plane parallel elements using spectrophotometers over the spectral range 190 nm to 25 μm.

The transmittance τ and the reflectance ρ of optical components are generally divided into two parts as follows:

$$\tau = \tau_r + \tau_d \quad (1)$$

$$\rho = \rho_r + \rho_d \quad (2)$$

where

τ_r is the regular transmittance;

τ_d is the diffuse transmittance;

ρ_r is the regular reflectance;

ρ_d is the diffuse reflectance.

This International Standard applies only 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 International Standard is applicable to test specimens which are coated or uncoated optical components without optical power.

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.

IEC 60050-845:1987, *International Electrotechnical Vocabulary — Chapter 845: Lighting*

ISO 31-6:1992, *Quantities and units — Part 6: Light and related electromagnetic radiations*

ISO 9211-1:1994, *Optics and optical instruments — Optical coatings — Part 1: Definitions*

ISO 9211-2:1994, *Optics and optical instruments — Optical coatings — Part 2: Optical properties*

ISO 10110-8:1997, *Optics and optical instruments — Preparation of drawings for optical elements and systems — Part 8: Surface texture*

3 Terms and definitions

For the purposes of this International Standard, the terms and definitions given in ISO 31-6, ISO 9211-1 and the following (which are given in IEC 60050-845) apply.

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

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

3.3
internal transmittance
ratio of the radiation flux reaching the internal exit surface of the layer to the flux that enters into the layer after crossing the entry surface

3.4
reflectance
〈for incident radiation of given spectral composition, polarization and geometrical distribution〉 ratio of the reflected radiant or luminous flux to the incident flux in the given conditions

3.5
regular reflectance
specular reflectance
ratio of the regularly reflected part of the whole reflected flux to the incident flux

4 Symbols and units

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

λ wavelength, expressed in nanometres

i angle of incidence, expressed in degrees

p, s state of polarization

τ transmittance

τ_r regular transmittance

τ_i internal transmittance

ρ reflectance

ρ_r regular reflectance

5 Test specimen

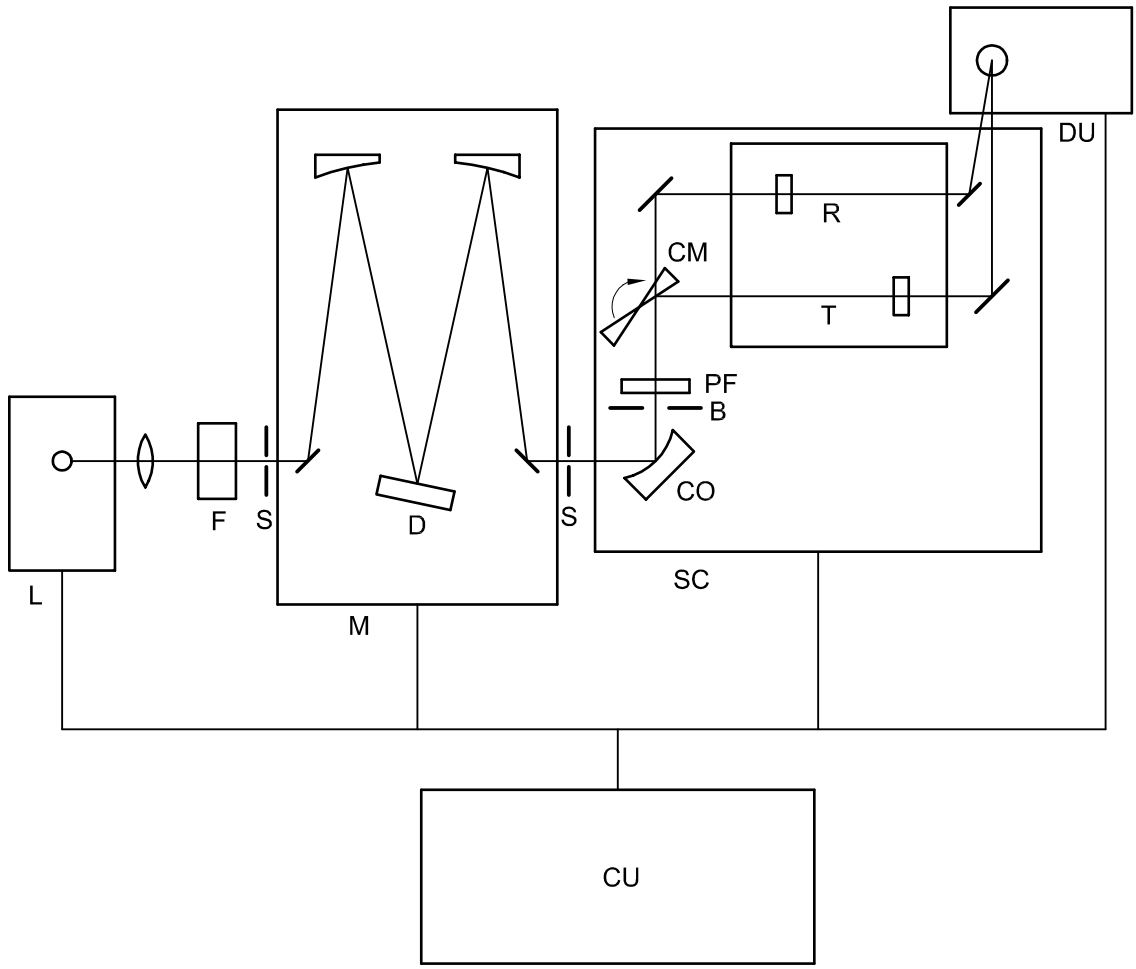
Storage, cleaning and preparation of a test specimen shall be carried out in accordance with the instructions of the manufacturer on the test specimen for normal use.

Wavelength, angle of incidence and state of polarization shall correspond to those specified by the manufacturer for the use of the test specimen.

6 Measuring apparatus

For the measurement specified in this International Standard, a spectrophotometer is required. Figure 1 shows an example of a double beam, dispersion type spectrophotometer. It consists of a light source, a monochromator, a specimen compartment, a detector unit and a control unit.

Details of the apparatus are described in annex A.



Key

L	Light source
F	Filter box
S	Slit
D	Dispersive element
M	Monochromator
SC	Specimen compartment
CO	Collecting optics
B	Baffle
PF	Polarization filter
CM	Chopper mirror
T	Test beam
R	Reference beam
DU	Detector unit
CU	Control unit

Figure 1 — Standard arrangement of a spectrophotometer

7 Test conditions

7.1 General

The light source, divergence of beam, beam diameter on the specimen, wavelength, spectral resolution, stepping interval, incident angle, detector and numerical correction shall be selected and documented.

7.2 Light source

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

NOTE The state of polarization of the radiation reaching the detector may be affected by reflection on components in the reference/sample paths. It is suggested to rotate the sample in its incidence plane to check for polarization effects.

The beam diameter on the specimen shall be larger than 1 mm. On the surface of the specimen 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 two. The beam diameter and the beam divergence (see also 9.9) shall be documented.

7.3 Monochromator

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

Optics for blocking out higher order diffraction light shall be documented.

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

The type of spectrophotometer (single or double beam, dispersion or Fourier-transform) shall be documented.

7.4 Detection system

An appropriate detector for the measuring spectral region shall be selected and documented. In the case of a dispersion type spectrophotometer, a lock-in detection technique is frequently used and a light 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 [1].

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

7.5 Numerical correction

Numerical correction can include spectral correction, averaging, smoothing, calibration of photometric linearity and others.

Spectral correction can be made referring to an appropriate wavelength standard (see 9.2). Random noise can be reduced by averaging or smoothing. Averaging can be made by repeating measurement or increasing sampling time. Smoothing can be made by averaging data in the finite spectral bandwidth after measurement, although it reduces spectral resolution. Sampling time and smoothing factors shall be documented.

For details on the calibration of photometric linearity, see 7.4.

Calibration of the spectrophotometer can be done by measuring the transmittance of a reference sample (standard) using the method given in 8.2.1. A reference sample for the transmittance from ultraviolet to near infrared region shall be an accurately parallel plate of fused silica with P2 grade surface specified in ISO 10110-8. Accuracy and

repeatability of the transmittance of this reference sample is from $\pm$ 0,02 % to $\pm$ 0,5 % including photometric noise. Other standard reference materials which are checked at an accredited laboratory may be used.

8 Test procedure

8.1 Measurement of reflectance

8.1.1 General

Either of the two types of measurements of reflectance, a direct method or a relative method, shall be chosen.

The incident angle shall be selected according to the manufacturer's instruction. Reflectance of normal incidence cannot usually be measured and the incident angle from 5° to 15° instead of 0°, which shall be documented, is used. In the case of an incident angle other than 0°, the reflectance depends on the state of polarization of the incident light, 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 2 shows two methods of the direct measurement of reflectance. In Figure 2 a), the reflected flux Φ_1 without a specimen is measured, and then the reflected flux Φ_2 with the specimen is measured after changing the optical arrangement as in Figure 2 b) and c). The regular reflectance of the specimen is given as

$$\rho_\mathrm{r} = \frac{\Phi_2}{\Phi_1} \tag{3}$$

[in the case of an arrangement as shown in Figure 2 b)]

$$\rho_\mathrm{r} = \sqrt{\frac{\Phi_2}{\Phi_1}} \tag{4}$$

[in the case of an arrangement as shown in Figure 2 c)]

irrespective of the magnitudes of the reflectance of the reference mirror and other optics.

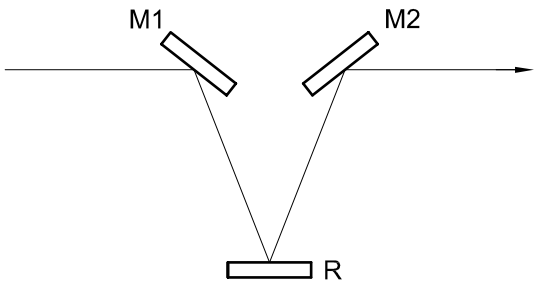
8.1.3 Relative measurement of regular reflectance

The relative measurement is easier than the direct measurement. An example of a reference sample for the reflectance is an aluminum mirror or a fused silica plate with a wedge angle, polished smoothly and kept clean. The successive measurements of the reflected flux of the reference sample Φ_ref and that of a specimen Φ_s are made using the arrangement of Figure 2 a). Then the regular reflectance of the specimen is given as

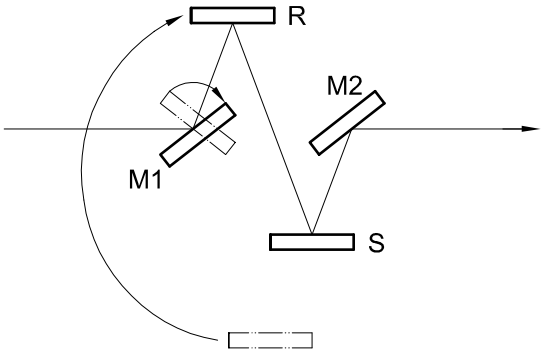
$$\rho_\mathrm{r} = \frac{\Phi_\mathrm{s}}{\Phi_\mathrm{ref}} \times \rho_\mathrm{ref} \tag{5}$$

where ρ_ref is the regular reflectance of the reference sample.

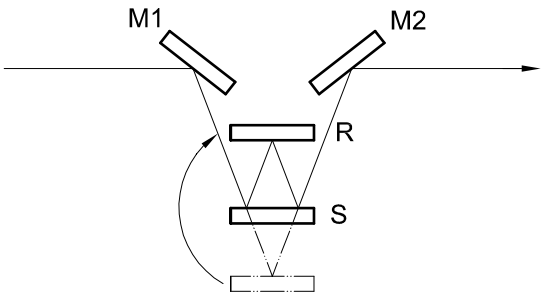
The value ρ_ref is calibrated separately by the direct method given in 8.1.2. For a low reflectance specimen such as an anti-reflection coated or uncoated glass plate, the relative measurement is recommended. In such a case, fused silica plate is used as the reference sample for the region from ultraviolet to 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.



a) Measurement of the reference



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



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

Key

M1	Mirror 1
M2	Mirror 2
R	Reference mirror
S	Specimen

Figure 2 — Direct measurement of reflectance

Other standard reference materials for which reliable refractive index data is available may be used and will be required for wavelengths longer than 3 μm. In case of any doubt about refractive index data, the material shall be calibrated by an accredited laboratory.

In the case of measurement of the regular reflectance of a transparent glass plate, for example, reflection from the second surface shall be eliminated. Reflection from the second surface causes a significant error, especially for a low reflectance specimen. One method of eliminating this reflection is to make the specimen wedge-shaped; the other method is to grind the second surface and coat it with absorbing paint.

In the case of 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.

8.2 Measurement of transmittance

8.2.1 Regular transmittance

For the measurement of regular transmittance, the flux with and without a specimen is measured. The ratio of the regularly transmitted flux with the specimen Φ_{w} to that without the specimen Φ_{wo} gives the regular transmittance τ_{r} as follows:

$$\tau_{\mathrm{r}} = \frac{\Phi_{\mathrm{w}}}{\Phi_{\mathrm{wo}}} \tag{6}$$

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

8.2.2 Internal transmittance of the 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 the specimen of 1 cm thickness is given as follows:

$$\tau_{\mathrm{i}} = \frac{\tau_{\mathrm{r12}}}{\tau_{\mathrm{r2}}} \tag{7}$$

The above equation is valid when internal multiple-reflection is ignored. For the specimen of very low transmittance, that is, of high absorptance, internal multiple-reflection can be ignored because multiple-reflected light is almost absorbed. For the specimen of high transmittance, internal multiple-reflection can also be ignored because the intensities of the multiple-reflected and transmitted light are almost the same for both τ_{r2} and τ_{r12}.

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

9 Main error factors

9.1 General

The main error factors of the spectrophotometer are wavelength accuracy, wavelength reproducibility, spectral resolution, fluctuation of incident flux (light source), parallelism of specimen, stray light, linearity of detection system, misalignment of specimen, baseline reproducibility and beam divergence. They are briefly described in 9.2 to 9.9; however, it is not possible to indicate their combination for the evaluation of measurement uncertainty in a general case.

The typical values of overall photometric accuracy and reproducibility of the spectrophotometer are $\pm$ 0,3 % and $\pm$ 0,1 % in the ultraviolet and visible region (190 nm to 780 nm), and $\pm$ 1 % and $\pm$ 0,5 % in the infrared region (780 nm to 25 μm).

9.2 Wavelength accuracy, reproducibility and spectral resolution

Wavelength accuracy and reproducibility of the typical monochromator is around 0,2 nm and 0,1 nm in the ultraviolet and visible regions, around 1 nm and 0,5 nm in the near infrared region (780 nm to 2,5 μm), and from 1 cm^{-1} to

5 cm^{-1} and from 0,5 cm^{-1} to 5 cm^{-1} in the middle infrared region (2,5 μm to 25 μm). The typical value of the spectral resolution is 0,1 nm in the ultraviolet to near infrared region, and from 0,1 cm^{-1} to 5 cm^{-1} in the middle infrared region.

Wavelength scale can be calibrated by several wavelength standards such as spectral lamps and a holmium and a didymium glass plate in the visible and near infrared regions. In the middle infrared region, absorption spectra of polystyrene, indene, toluene, trichlorobenzene, etc. and rotation-vibration spectra of CO_2, CO, HCl, etc. are used.

Wavelength accuracy, reproducibility and spectral resolution shall be documented.

9.3 Fluctuation of incident flux

Typical fluctuation of the light 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 may also be used.

9.4 Parallelism of specimen

In the case of measurement of regular reflectance, the reflectance of a single surface is usually of major concern. Reflection from the other surface shall be eliminated for the measurement of an anti-reflection coating or a semi-transparent mirror, for example.

In the case of measurement of transmittance, the measured value depends on the wedge angle between the first and second surfaces. When the specimen is parallel, multiple-reflected beams from both surfaces enter 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 beams from both surfaces do not enter the detector. When a large wedge angle is used, it shall be documented.

NOTE See also ISO 9211-2:1994, 8.1.2, 8.2.2 and annex A.

9.5 Stray light

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

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

9.6 Linearity of detection system

Calibration and testing of linearity of the detection system is described in 7.4. Typical nonlinearity is 1 %.

9.7 Misalignment of specimen

Influence of misalignment of the specimen depends on the characteristics of the detector. When the test beam is shifted or tilted by the misalignment of the specimen, the angle or the position of the incident beam changes and the sensitivity of the detector may change. To eliminate this error and to assure reproducibility of the measured value, an appropriate positioning device shall be used [also see d) of A.2]. In the case of measurement of transmittance, the reflected light from the specimen causes stray light or a ghost. To reduce it, the specimen shall be tilted slightly in relation to its nominal position (perpendicular to the beam).

Thick and/or high index specimens 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.

In Fourier-transform type spectrometers, reflection from a specimen positioned perpendicularly or near perpendicularly to the beam may be processed by the instrument leading to erroneous results. A significant tilt of the specimen may be needed to ensure that this is not a problem.

9.8 Baseline reproducibility

Reproducibility and noise level of the baseline (100 % transmittance level) is from $\pm$ 0,3 % to $\pm$ 0,5 % in the visible and near infrared spectral regions.

9.9 Beam divergence

Typical beam divergence of the spectrophotometer is less than 5°. The measurement is reliable when most beams enter the detector.

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 on the test specimen

   1) Manufacturer of specimen;

   2) Specifications of manufacturer for storage and cleaning;

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

   4) Part identification code, date of production.

c) Information on the test

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

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

   3) Parameters of the detection system (scanning speed, sampling time, averaging and smoothing factors);

   4) Error budget [wavelength accuracy, wavelength reproducibility, fluctuation of incident flux (light source), parallelism of specimen, stray light, detector linearity, baseline reproducibility, error of reference sample];

   5) Photometric accuracy and reproducibility;

   6) Environmental conditions (temperature, degree of cleanness when cleanroom is used, purge gas).

d) Results

   Graph and/or table of spectral characteristics of specimen.

e) A reference to this International Standard, ISO 15368.

Annex A
(informative)

Spectrophotometers

A.1 General

This annex describes spectrophotometers used in the spectral range between 190 nm and 25 μm.

A.2 Dispersion type spectrophotometer

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

a) **Light source**

A D_2 (deuterium) lamp is used in the ultraviolet spectral region (190 nm to 350 nm), a tungsten (halogen) lamp in the ultraviolet, visible and infrared spectral regions (250 nm to 3,0 μm) and a Globar (SiC) or helically wound nichrome wire in the infrared spectral region (2,5 μm to 25 μm).

b) **Monochromator**

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

A dispersive element is a diffraction grating (190 nm to 25 μm), a glass prism (400 nm to 2,0 μm), a fused silica prism (190 nm to 3,0 μm) or a KBr prism (2,0 μm to 25 μm). For blocking the higher order light diffracted from the 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 light, a double monochromator is recommended.

c) **Specimen compartment**

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

1) Collecting optics

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

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.

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 220 nm and 2,3 μm, a polarizing prism made of birefringent crystal is available. In the spectral range between 280 nm and 2,0 μm, a polarization filter made of a sheet polarizer is convenient. In the middle infrared region, a wire grid polarizer can be used.

4) Test and reference beam

The spectrophotometer is divided into two types; a single beam and a double beam (i.e. a test and a reference beam). The double beam type has the advantage of compensating the fluctuation of the light source.

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

5) Baffle

In order to shape and collimate the beam and to eliminate stray light, several baffles are 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 specimen or, if possible, on the detector.

d) **Detector unit**

A photomultiplier or a silicon photodiode is used in the ultraviolet and visible regions, a silicon photodiode, a germanium photodiode or a PbS cell cooled to reduce thermal noise in the near-infrared region and a vacuum thermopile in the middle infrared region.

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 ground glass is installed in front of the detector. The magnitude of the signal is then largely reduced.

e) **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 spectrometer

In the spectral range between 2,5 μm and 25 μm (the wave number range 4 000 cm^{-1} and 400 cm^{-1}), a Fourier-transform type spectrometer is widely used. A Globar or a high pressure Hg lamp is used as a source and a TGS (triglicine sulfate) pyroelectric cell or a MCT (Hg-Cd-Te) photoconductive cell cooled by liquid N_2 is used as a detector.

Interference fringes (interferograms) obtained with an interferometer (typically Michelson type) are Fourier-transformed in order to obtain spectra.

Annex B
(informative)

Refractive index of synthetic fused silica

The refractive index of synthetic fused silica at 20 °C in the spectral range from 213,9 nm to 3,7067 μm can be calculated by use of the following equation:

$$n^2 - 1 = \frac{0{,}6\ 961\ 663\lambda^2}{\lambda^2 - (0{,}0\ 684\ 043)^2} + \frac{0{,}4\ 079\ 426\lambda^2}{\lambda^2 - (0{,}1\ 162\ 414)^2} + \frac{0{,}8\ 974\ 794\lambda^2}{\lambda^2 - (9{,}896\ 161)^2} \quad \text{(B.1)}$$

where λ is expressed in micrometres [2].

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