INTERNATIONAL STANDARD

ISO 23946

First edition
2020-11

Fine ceramics (advanced ceramics, advanced technical ceramics) — Test methods for optical properties of ceramic phosphors for white light-emitting diodes using a gonio-spectrofluorometer

Reference number
ISO 23946:2020(E)

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Published in Switzerland

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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

This document was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

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

White light-emitting diode (LED)-based solid-state lighting (SSL) has been widely used for a variety of applications as an alternative for incandescent and fluorescent lamps. In the beginning, white LEDs (comprising blue LEDs and yellow phosphors) became popular as backlight sources for small-size liquid-crystal displays (LCDs) used in mobile phones and digital cameras. These were followed by white LEDs (consisting of blue LEDs combined with green and red phosphors) applied to backlight sources for large-area LCDs. Subsequently, LED lamps have been commercialized for general lighting, replacing conventional luminaires and capitalising on their advantages, such as compactness, high luminous efficiency, high brightness below 0 °C or higher ambient temperatures, long life and controllability of light intensity and colour temperature.

Optical performance of a phosphor material for use in a white LED is one of the most important factors influencing the performance of the white LED. Accordingly, it is of great importance not only for researchers and manufacturers of phosphors for use in white LEDs but also for researchers and manufacturers of white LED devices to evaluate optical properties of the phosphors in a well-established manner. Photoluminescence quantum efficiency is one of the key optical parameters of phosphors for use in white LEDs and has been measured extensively by using an integrating sphere-based absolute method.

ISO 20351 was developed in accordance with the demand for standardizing the test method of internal quantum efficiency of phosphors using an integrating sphere. This standard test method has the advantage of short measurement time and being available to those with no expertise in precise optical measurement. Despite their importance in terms of the performance of ceramic phosphor products, however, external quantum efficiency and absorptance are out of the scope of ISO 20351 due to insufficient understanding of the source of variation in these measurement values.

This document provides the absolute measurement methods of external quantum efficiency and absorptance as well as internal quantum efficiency and related optical properties for ceramic phosphors for use in white LEDs using a gonio-spectrofluorometer. This equipment is regarded as one of the variations of a gonio-reflectometer commonly used to evaluate optical properties of material surfaces.

In this document, measurement conditions and procedures, which can affect the measurement values, are described in detail, helping those who address the high-performance phosphors for competitive SSL products to obtain the proper information on their competitiveness.

This document can also be adopted to phosphors used in non-white LEDs, for example green, orange, pink and purple.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Test methods for optical properties of ceramic phosphors for white light-emitting diodes using a gonio-spectrofluorometer

1 Scope

This document specifies a method for use of a gonio-spectrofluorometer to measure internal quantum efficiency, external quantum efficiency, absorptance, luminescent radiance factor and relative fluorescence spectrum of ceramic phosphor powders which are used in white light-emitting diodes (LEDs) and emit visible light when excited by UV or blue light.

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 20351, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Absolute measurement of internal quantum efficiency of phosphors for white light emitting diodes using an integrating sphere*

CIE S 017/E, *International Lighting Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 20351 and CIE S 017/E 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
gonio-spectrofluorometer
apparatus measuring the observation angle dependence of the spectral distribution of fluorescent light or scattered light emitted by a sample irradiated on its surface by a monochromatic light

3.2
in-plane
<optical radiation> emitted or reflected, with a propagation vector located in a plane of incidence

3.3
out-of-plane
<optical radiation> emitted or reflected, with a propagation vector not located in a plane of incidence

4 Spherical coordinate system

The coordinate system used in gonio-spectrofluorometry shall be a spherical coordinate system (r, θ, ϕ). In a gonio-spectrofluorometer, the plane including the sample surface shall be taken as the horizontal plane and the centre of the surface of the sample shall be taken as the origin. The radial distance r

of the observation point shall be held constant during the measurement. The geometrical parameters of measurement are defined by the angle of incidence θ_i, the zenith angle of observation θ_r and the azimuth angle of observation ϕ_r. The vertical axis is defined as the direction where $\theta_i = \theta_r = 0°$, and the plane of incidence is defined as $\phi_r = 0°$.

5 Measurement apparatus

5.1 Apparatus configuration

The apparatus comprises elements including a light source unit, a sample unit, a detection unit, a rotational positioning unit, an enclosure and a signal/data processing unit. Figure 1 illustrates the typical measurement apparatus configuration.

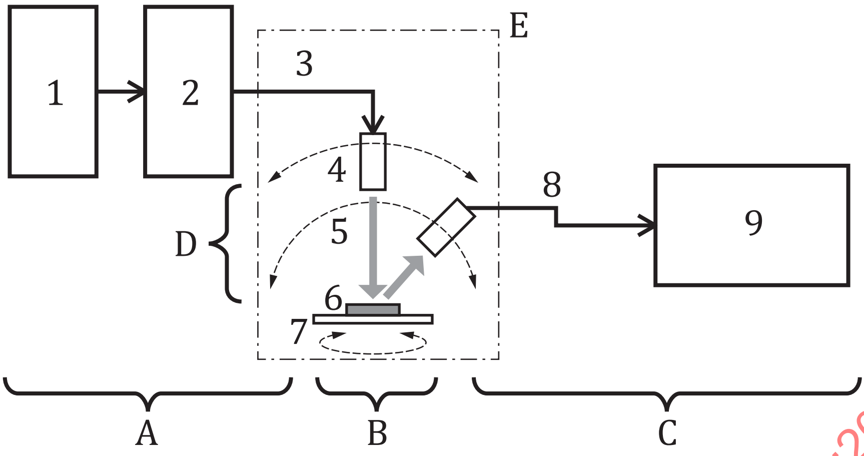
The light source unit generates monochromatic excitation light and comprises a white light source, a power supply for the white light source, a focusing optical system, a wavelength selection unit (monochromator for the white light source) and an optical system for irradiation. A collimated laser beam can also be used as the monochromatic light source.

The sample unit comprises a cell, a diffuse reflectance standard and a sample stage.

The detection unit comprises a directing optical system for collecting light, a spectrometer, a detector and an amplifier.

Example measurement configuration is illustrated in Figure 2, where the geometrical parameters are defined in Clause 4. The rotational positioning unit for measuring in-plane spatial distribution comprises a mechanism for setting the angle of incidence and a mechanism for setting the zenith angle of observation. When out-of-plane spatial distribution is measured, the rotational positioning unit also includes mechanism for setting the azimuth angle of observation. When gonio-spectrofluorometric measurement is performed with a certain fixed angle of incidence, a non-adjustable optical system for irradiating incident beam onto the centre of a sample surface may be used. For measuring in-plane spatial distribution, the incident optical axis is located in the same plane as that where the observation optical axis is rotating by the mechanism for setting the zenith angle of observation. To prevent the mechanism for setting the zenith angle-of-observation from interfering with the mechanism for setting the angle-of-incidence, each can be given radial distance that differs substantially from the other. Alternatively, one or both of these mechanisms can be provided with a supplemental positioning mechanism for preventing collision.

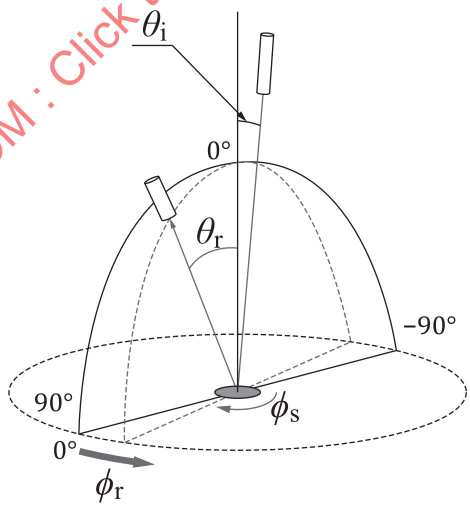
Each component incorporated inside the enclosure should have a matte black surface to reduce stray light.



Key

A	light source unit	1	light source
B	sample unit	2	spectrometer
C	detection unit	3	optical system for irradiation (optical fibre probe)
D	rotational positioning unit	4	mechanism for setting angle of incidence
E	enclosure	5	mechanism for setting zenith angle of observation
		6	sample (cell)
		7	sample stage
		8	directing optical system (optical fibre probe)
		9	array spectrometer

Figure 1 — Typical measurement apparatus configuration



Key

θ_r zenith angle of observation

θ_i angle of incidence

ϕ_r azimuth angle of observation

ϕ_s sample rotation angle

Figure 2 — Example configuration of each geometrical parameter

5.2 Light source unit

The spectral width of the excitation light is limited by the spectrometer. The half-width of the excitation light spectrum is preferably 15 nm or less.

The excitation light passes through an optical system for irradiation and irradiates a sample or a diffuse reflectance standard. One example of an optical system for irradiation is an optical fibre probe. One end of the fibre probe is attached to the exit slit of the monochromator and the monochromated light is emitted from the other end. A focusing optics attached to the end of the fibre probe provides a circular, nearly circular or oval-shaped beam of light to illuminate the sample surface.

The optical system for irradiation should be designed to optimize the size of illuminating area on the sample and the diffuse reflectance standard for detecting scattered light and fluorescence efficiently.

5.3 Sample unit

5.3.1 Cell

The area of a sample shall be substantially larger than the area irradiated by the excitation light, and the thickness of a sample in the normal direction shall be at least 2 mm.

A cell shall be made of chemically and physically stable material which does not contaminate the sample inside and can be used in conjunction with a cell adapter provided for a sample stage.

When a less absorptive sample is measured, a cell with a transparent window at its bottom can be used to examine fluorescence or scattered light emitting from the bottom of the cell (see Annex A).

The top surface of the cell shall have a cover glass or a lid to prevent a sample powder from dispersing and contaminating its surroundings during transport or preparation for installation. However, the cover glass or a lid shall be removed during the gonio-spectrofluorometric measurement to expose the sample surface.

5.3.2 Diffuse reflectance standard

A diffuse reflectance standard is used as a reference standard in gonio-spectrofluorometric measurement for calibrating a spectral radiance factor or bi-directional reflectance distribution function (BRDF) in the wavelength range including the excitation light wavelength and the fluorescence light wavelength. The diffuse reflectance standard used shall be an item with a diffuse reflectance of 90 % or greater and a spatial distribution of diffused light close to Lambertian at an incident angle from 0° to 30°, examples of which include a sintered polytetrafluoroethylene material. A secondary diffuse reflectance standard (working standard) can also be used, where the working standard provides calibration for a spectral radiance factor or BRDF using a gonio-spectrofluorometer or another spatial light distribution measurement apparatus, based on a diffuse reflectance standard used to calibrate a spectral radiance factor or BRDF. The working standard used shall be an item with a diffuse reflectance of 90 % or greater and a spatial distribution of diffused light close to Lambertian at an incident angle from 0° to 30°, an example of which is a cell filled with pressed barium sulfate powder.

5.3.3 Sample stage

A sample stage allows a cell to be placed with a cell adapter, if any, at the centre portion of the stage such that the sample surface is always kept horizontal. The sample stage is preferably provided with an automatic or manual mechanism rotating about the vertical axis for setting a sample rotational angle ϕ_s when a surface of a ceramic phosphor sample is not confirmed in advance to be substantially uniform with respect to the sample rotation.

5.4 Detection unit

5.4.1 Directing optical system

Fluorescence light and scattered light from the sample surface is directed through a directing optical system to a spectrometer. The directing optical system shall have sufficient transmissivity over the entire measured spectral range. An optical fibre probe is an example of a directing optical system. A focusing optics is preferably attached to an end of the fibre probe so as to direct the collected light efficiently to the fibre probe.

5.4.2 Spectrometer and detector

This equipment converts light directed through the directing optical system to electrical signals proportional to the intensity spectrum of the light. A photomultiplier or a CCD detector, with sufficient sensitivity over the measured spectral range, is an example of a detector. An array spectrometer is a typical example of a system combining a spectrometer and a detector, but a wavelength scanning spectrometer can also be used.

5.4.3 Amplifier

This device amplifies the electrical signal from the detector for data processing.

5.5 Rotational positioning unit

5.5.1 Mechanism for setting angle of incidence

This unit is provided with an arm which holds the optical system for irradiation and a rotating mechanism for the arm to vary the angle of incidence while the centre of irradiation remains constant. The rotating mechanism may be either automatic or manual, but in either case setting of the angle of incidence should be sufficiently reproducible.

5.5.2 Mechanism for setting zenith angle of observation

This unit is provided with an arm which holds the directing optical system, and a rotating mechanism for the arm to vary the zenith angle of observation at defined angular intervals. Its rotational axis shall be a horizontal axis which passes through the origin, namely the centre of the irradiated area on the sample surface with excitation light. The rotating mechanism may be either automatic or manual, but in either case setting of the zenith angle of observation should be sufficiently reproducible.

5.5.3 Mechanism for setting azimuth angle of observation

This mechanism is used to set an azimuth angle of observation ϕ_r when out-of-plane spatial distribution is measured. A mechanism for either setting a zenith angle of observation or setting an angle of incidence is designed to rotate with the angle ϕ_r about the vertical axis. The rotating mechanism may be either automatic or manual, but in either case setting of the azimuth angle of observation should be sufficiently reproducible.

5.6 Enclosure

The enclosure is covered with a blackout curtain and houses internally the optical system for irradiation, the directing optical system, the sample unit and the rotational positioning unit. The blackout curtain used has a substantial light-blocking capacity for outside light environment, and its inner surface is covered with a dust-proof and anti-static processed matte black material with low diffuse reflectance to suppress stray light and prevent dust during the gonio-spectrofluorometric measurement.

5.7 Signal and data processing unit

This unit separates and processes signals required for measurement, outputs light intensity as a photon number or energy for each angle of observation (zenith angle of observation θ_r and azimuth angle of observation ϕ_r) and measured wavelengths, and saves the associated data.

6 Calibration, inspection and maintenance of measurement apparatus

6.1 General

Measuring equipment should be calibrated in the proper manner for accurate optical measurement. In addition, the equipment as well as its accessories should be maintained to keep its condition optimal. The quality control manager should make sure to undertake a regular checking procedure according to the manufacturer's suggestions. Routine factory checking by the manufacturer is also desirable.

6.2 Wavelength calibration of light source unit

When using a monochromated light source, use a monochromator whose wavelength is calibrated with the line source (e.g. a low-pressure mercury lamp) of known wavelength.

When using a laser light source, verify the wavelength emitted using a spectrometer or wavemeter calibrated separately for wavelength.

6.3 Cells

Handle cells carefully to avoid damage. Replace damaged cells with new items.

6.4 Diffuse reflectance standard

Radiance factor or BRDF of the diffuse reflectance standard at excitation wavelength should be calibrated periodically.

6.5 Wavelength calibration of detection unit

Use a spectrometer whose wavelength is calibrated with the line source (e.g. a low-pressure mercury lamp) of known wavelength.

6.6 Spectral responsivity calibration

The relative spectral responsivity of the entire detecting unit should be calibrated using a spectral irradiance standard light source. All measurement spectra should be corrected based on the relative spectral responsivity calibration results.

6.7 Rotational positioning unit

Check periodically that the mechanisms for setting angle of incidence and zenith angle of observation, as well as the mechanism for setting azimuth angle of observation for measuring out-of-plane spatial distribution, are operating correctly. Also check that the centre of the irradiated area on the sample surface is always kept at the origin of the rotational positioning unit.

7 Samples

7.1 Storage and pre-processing

Phosphor samples shall be stored appropriately according to their properties and pre-processed as necessary. Samples can normally be stored at ambient temperature in a desiccator; however, samples which react with airborne moisture and samples which can be degraded by UV or visible light should be stored with an inert gas under seal using a glove box or a coloured bottle.

Samples which absorb moisture readily should be dried before measurement in a vacuum dry oven at a non-deteriorating temperature.

7.2 Filling cells with samples

Overfill the hollow of the cell with an excessive amount of sample, press it down with a flat plate and scrape off the excess. The sample surface thus prepared should not be pressed with a flat plate, which can induce specularity on the surface. Until the cell is placed in the sample unit to be ready for measurement, cover the top with a lid to prevent dispersion of sample powder.

8 Measurement procedures

8.1 Measurement environment

Locate the measurement apparatus in an environment where ambient temperature can be maintained and avoid sudden temperature changes by measures such as locating the apparatus out of direct sunlight.

Handle and measure samples in a stable environment with an ambient temperature of 10 °C to 30 °C and a relative humidity of 20 % to 80 %. For hygroscopic samples and those with little durability, prepare a measurement environment suited to these characteristics, and complete measurement within as short a duration as possible. Turn on the measuring apparatus at least 30 min prior to the measurement.

8.2 In-plane spatial distribution measurement

8.2.1 Goniometric measurement of diffuse reflectance standard

8.2.1.1 Automatic measurement

Place the diffuse reflectance standard onto a sample stage in conjunction with a cell adapter (if any). Measure and record the angle of incidence. Next, in the measurement software, set the starting angle, ending angle and angle interval for the zenith angle of observation. Close the blackout curtain, verify that the dark count is sufficiently low and then begin measurement. After measurement is complete, save the spectral measurement data for all the varying zenith angles of observation.

8.2.1.2 Manual measurement

Place the diffuse reflectance standard onto a sample stage in conjunction with a cell adapter (if any). Measure and record the angle of incidence. Next, set each of the setting mechanisms for the initial zenith angle of observation. Close the blackout curtain, verify that the dark count is sufficiently low and then begin measurement. After measurement is complete, perform the same measurement for the next angle setting. After measurement is complete for all angles of spatial light distribution, save the spectral measurement data for all the varying zenith angles of observation.

8.2.2 Gonio-spectrofluorometric measurement of phosphor sample

8.2.2.1 Automatic measurement

Secure a cell filled with a phosphor sample onto a sample stage in conjunction with a cell adapter (if any). Verify that the angle of incidence is identical with that of the measurement of diffuse reflectance standard as described in 8.2.1.1 and record it. Next, in the measurement software, set the starting angle, ending angle and angle interval for the zenith angle of observation. Close the blackout curtain, verify that the dark count is sufficiently low and then begin measurement. After measurement is complete, save the spectral measurement data for all the varying zenith angles of observation.

8.2.2.2 Manual measurement

Secure a cell filled with a phosphor sample onto a sample stage in conjunction with a cell adapter (if any). Verify that the angle of incidence is identical with that of the measurement of diffuse reflectance standard as described in 8.2.1.2 and record it. Next, set each of the setting mechanisms for the initial zenith angle of observation. Close the blackout curtain, verify that the dark count is sufficiently low and then begin measurement. After measurement is complete, perform the same measurement for the next angle setting. After measurement is complete for all angles of spatial light distribution, save all the spectral measurement data.

8.3 Measurement of spatial light distribution with varying azimuth angle of observation

If scattered light or fluorescent light intensity is apparently dependent on the azimuth angle of observation, measurement should not be limited to in-plane spatial distribution as described in 8.2 but should be extended to out-of-plane spatial distribution. In that case, use the azimuth angle of observation setting mechanism to set an azimuth angle of observation for individual, defined angle intervals and repeat the measurement. First, set up a diffuse reflectance standard and follow the measurement procedures described in 8.2.1 for each azimuth angle of observation. Next, set up a phosphor sample and follow the measurement procedures described in 8.2.2 for each azimuth angle of observation.

8.4 Evaluation of surface uniformity with varying azimuth rotational angle of sample

A sample stage equipped with an automatic or manual rotation about the vertical axis allows the surface uniformity of the diffuse reflectance standard and phosphor sample to be evaluated. The azimuth rotational angle of the sample can be set to a certain angle interval and measurement can be performed as described in 8.2.1 and 8.2.2. In this instance, determine the mean of data from measurement with varying azimuth rotational angles of a sample made at the same zenith angle of observation and, when out-of-plane spatial distribution is measured, at the same zenith and azimuth angles of observation, and take measurement results for the associated directions of observation.

9 Calculation

9.1 Relative spectral distribution

9.1.1 Spectral responsivity and accumulation time corrections

Convert each measured scattered light spectrum of the diffuse reflectance standard, which is obtained as the data set of detection wavelength-dependent signal intensities for each observation angle, to corresponding spectral quantity proportional to spectral irradiance by using spectral responsivity correction data as described in 6.6 and data accumulation time (if different for each angle of observation).

Convert each measured scattered light or fluorescence spectrum of the phosphor sample, which is obtained as the data set of detection wavelength-dependent signal intensities for each observation angle, to corresponding spectral quantity proportional to spectral irradiance by using spectral responsivity correction data as described in 6.6 and data accumulation time (if different for each angle of observation).

9.1.2 Mean spectrum for varying azimuth rotational angle of sample

When measuring each spectrum with varying azimuth rotational angle of a sample as described in 8.4, determine its mean spectrum using Formulae (1) and (2) or Formulae (3) and (4).

For in-plane spatial distribution measurement as described in 8.2:

$$\overline{E_R}(\lambda,\lambda_{ex},\theta_i,\theta_r)=\frac{\sum_{n=0}^{N-1}E_R(\lambda,\lambda_{ex},\theta_i,\theta_r,\phi_{s,n})}{N} \quad (1)$$

$$\overline{E_S}(\lambda,\lambda_{ex},\theta_i,\theta_r)=\frac{\sum_{n=0}^{N-1}E_S(\lambda,\lambda_{ex},\theta_i,\theta_r,\phi_{s,n})}{N} \quad (2)$$

where

E_R is the spectral intensity of scattered light of diffuse reflectance standard;

$\overline{E_R}$ is the averaged spectral intensity E_R;

E_S is the spectral intensity of scattered light and fluorescence of the sample;

$\overline{E_S}$ is the averaged spectral intensity E_S;

λ_{ex} is the excitation wavelength;

λ is the detection wavelength;

θ_i is the angle of incidence;

θ_r is the zenith angle of observation;

$\phi_{s,n}$ is the nth azimuth rotational angle of a sample (n = 0, 1, ..., N-1);

N is the number of spectrum taken for varying azimuth rotational angle of a diffuse reflectance standard or a sample.

For measurement of spatial light distribution with varying azimuth angle of observation as described in 8.3:

$$\overline{E_R}(\lambda,\lambda_{ex},\theta_i,\theta_r,\phi_r)=\frac{\sum_{n=0}^{N-1}E_R(\lambda,\lambda_{ex},\theta_i,\theta_r,\phi_r,\phi_{s,n})}{N} \quad (3)$$

$$\overline{E_S}(\lambda,\lambda_{ex},\theta_i,\theta_r,\phi_r)=\frac{\sum_{n=0}^{N-1}E_S(\lambda,\lambda_{ex},\theta_i,\theta_r,\phi_r,\phi_{s,n})}{N} \quad (4)$$

where ϕ_r is the azimuth angle of observation.

9.2 Conversion to photon number-based spectral distribution

In the case of measurement with fixed azimuth rotational angle of a sample, convert each corrected (9.1.1) energy-based spectral distribution to the corresponding photon number-based spectral distribution using Formulae (5) and (6):

$$E_R{}^{ph}=E_R\times\frac{\lambda}{hc} \quad (5)$$

$$E_S{}^{ph}=E_S\times\frac{\lambda}{hc} \quad (6)$$

where

$E_R{}^{ph}$ is the photon-based spectral intensity of scattered light of diffuse reflectance standard;

$E_S{}^{ph}$ is the photon number-based spectral intensity of scattered light and fluorescence of the sample;

λ is the detection wavelength;

h is the Planck constant;

c is the speed of light.

In the case of measurement with varying azimuth rotational angle of a sample, convert each corrected (9.1.1) and averaged (9.1.2) energy-based spectral distribution to the corresponding photon number-based spectral distribution using Formulae (7) and (8):

$$\overline{E_R{}^{ph}} = \overline{E_R} \times \frac{\lambda}{hc} \quad (7)$$

$$\overline{E_S{}^{ph}} = \overline{E_S} \times \frac{\lambda}{hc} \quad (8)$$

where

$\overline{E_R{}^{ph}}$ is the averaged photon number-based spectral intensity of scattered light of diffuse reflectance standard;

$\overline{E_S{}^{ph}}$ is the averaged photon number-based spectral intensity of scattered light and fluorescence of the sample.

9.3 Calculation of scattered light and fluorescence photon numbers

9.3.1 Scattered light photon number for diffuse reflectance standard

Use formulae tabulated in Table 1 for wavelength integration of the scattered light spectrum of a diffuse reflectance standard calculated on a photon number basis (9.2) in the wavelength range of the excitation light ($\lambda_{ex}-\Delta\lambda \le \lambda \le \lambda_{ex}+\Delta\lambda$) and calculate the scattered light photon number W at each observation angle.

Table 1 — Formulae for calculating the scattered light photon number of diffused reflectance standard

		Azimuth rotational angle of sample	
		Fixed	Variable
Azimuth angle of observation	Fixed	$W(\lambda_{ex},\theta_i,\theta_r,\phi_r)=\int_{\lambda_{ex}-\Delta\lambda}^{\lambda_{ex}+\Delta\lambda} E_R{}^{ph}(\lambda,\lambda_{ex},\theta_i,\theta_r,\phi_r)d\lambda$	$W(\lambda_{ex},\theta_i,\theta_r,\phi_r)=\int_{\lambda_{ex}-\Delta\lambda}^{\lambda_{ex}+\Delta\lambda} \overline{E_R{}^{ph}}(\lambda,\lambda_{ex},\theta_i,\theta_r,\phi_r)d\lambda$
	Variable	$W(\lambda_{ex},\theta_i,\theta_r)=\int_{\lambda_{ex}-\Delta\lambda}^{\lambda_{ex}+\Delta\lambda} E_R{}^{ph}(\lambda,\lambda_{ex},\theta_i,\theta_r)d\lambda$	$W(\lambda_{ex},\theta_i,\theta_r)=\int_{\lambda_{ex}-\Delta\lambda}^{\lambda_{ex}+\Delta\lambda} E_R{}^{ph}(\lambda,\lambda_{ex},\theta_i,\theta_r)d\lambda$

where

W	is the scattered light photon number of diffuse reflectance standard;
E_R^{ph}	is the photon number-based spectral intensity of scattered light of diffuse reflectance standard;
$\overline{E_R^{ph}}$	is the averaged photon number-based spectral intensity of scattered light of diffuse reflectance standard;
λ	is the detection wavelength;
λ_{ex}	is the excitation wavelength;
$\Delta\lambda$	is the half-width of excitation wavelength range;
θ_i	is the angle of incidence;
θ_r	is the zenith angle of observation;
ϕ_r	is the azimuth angle of observation.

9.3.2 Scattered and fluorescence photon numbers for phosphor sample

Calculate the scattered light photon number and fluorescence photon number at each observed angle based on the relative fluorescence spectrum of a phosphor sample calculated on a photon number-basis (9.2).

If the excitation wavelength range and the fluorescence wavelength range are substantially separated and can be easily separated on the spectrum, integrate the wavelengths in the respective wavelength ranges and determine the scattered light photon number S and the fluorescence photon number F using Formulae tabulated in Tables 2 and 3.

Table 2 — Formulae for calculating the scattered light photon number of phosphor sample

		Azimuth rotational angle of sample	
		Fixed	Variable
Azimuth angle of observation	Fixed	$S(\lambda_{ex},\theta_i,\theta_r,\phi_r)=\int_{\lambda_{ex}-\Delta\lambda}^{\lambda_{ex}+\Delta\lambda}\overline{E_S^{ph}}(\lambda,\lambda_{ex},\theta_i,\theta_r,\phi_r)\mathrm{d}\lambda$	$S(\lambda_{ex},\theta_i,\theta_r,\phi_r)=\int_{\lambda_{ex}-\Delta\lambda}^{\lambda_{ex}+\Delta\lambda}E_S^{ph}(\lambda,\lambda_{ex},\theta_i,\theta_r,\phi_r)\mathrm{d}\lambda$
	Variable	$S(\lambda_{ex},\theta_i,\theta_r)=\int_{\lambda_{ex}-\Delta\lambda}^{\lambda_{ex}+\Delta\lambda}\overline{E_S^{ph}}(\lambda,\lambda_{ex},\theta_i,\theta_r)\mathrm{d}\lambda$	$S(\lambda_{ex},\theta_i,\theta_r)=\int_{\lambda_{ex}-\Delta\lambda}^{\lambda_{ex}+\Delta\lambda}E_S^{ph}(\lambda,\lambda_{ex},\theta_i,\theta_r)\mathrm{d}\lambda$

Table 3 — Formulae for calculating the fluorescence photon number of phosphor sample

		Azimuth rotational angle of sample	
		Fixed	Variable
Azimuth angle of observation	Fixed	$F(\lambda_{ex},\theta_i,\theta_r,\phi_r)=\int_{\lambda_1}^{\lambda_2}\overline{E_S^{ph}}(\lambda,\lambda_{ex},\theta_i,\theta_r,\phi_r)\mathrm{d}\lambda$	$F(\lambda_{ex},\theta_i,\theta_r,\phi_r)=\int_{\lambda_1}^{\lambda_2}E_S^{ph}(\lambda,\lambda_{ex},\theta_i,\theta_r,\phi_r)\mathrm{d}\lambda$
	Variable	$F(\lambda_{ex},\theta_i,\theta_r)=\int_{\lambda_1}^{\lambda_2}\overline{E_S^{ph}}(\lambda,\lambda_{ex},\theta_i,\theta_r)\mathrm{d}\lambda$	$F(\lambda_{ex},\theta_i,\theta_r)=\int_{\lambda_1}^{\lambda_2}E_S^{ph}(\lambda,\lambda_{ex},\theta_i,\theta_r)\mathrm{d}\lambda$

where

S is the scattered light photon number of the phosphor sample;

F is the fluorescence photon number of the phosphor sample;

$E_S{}^{ph}$ is the photon number-based spectral intensity of scattered light and fluorescence of the phosphor sample;

$\overline{E_S{}^{ph}}$ is the averaged photon number-based spectral intensity of scattered light and fluorescence of the phosphor sample;

λ is the detection wavelength;

λ_{ex} is the excitation wavelength;

λ_1 is the short wavelength limit of the fluorescence wavelength range;

λ_2 is the long wavelength limit of the fluorescence wavelength range;

$\Delta\lambda$ is the half-width of excitation wavelength range;

θ_i is the angle of incidence;

θ_r is the zenith angle of observation;

ϕ_r is the azimuth angle of observation.

If the excitation light wavelength range and the fluorescence wavelength range are adjoining, and wavelength integration according to formulae tabulated in Tables 2 and 3 may unintentionally incorporate other components, use the relative fluorescence spectrum and the scattered light spectrum to separate the excitation light scattering component and the fluorescence component. The following methods are examples for separation and extraction of the fluorescence component.

Method 1: apply a suitable scale factor to the excitation light spectrum obtained in 8.2.1 and subtract the resulting spectrum from that obtained in 8.2.2 so as to obtain a smooth subtracted spectrum at the vicinity of the excitation light. Irregularities in the vicinity of the excitation wavelength can be removed, for example, by linear fitting of data points adjacently outside of the spectral area of irregularities.

Method 2: approximate the baseline in the vicinity of the excitation light as a linear plot.

9.4 Average of scattered light or fluorescence photon number for variable azimuth angle of observation

When out-of-plane spatial distribution is measured, calculate the mean for measurement of the zenith angle of observation as in the case of the azimuth angle of observation set for each defined angular range. At this time, interpolate an estimate of values for dead angles produced near the incident direction, with such an estimate based on values at the nearby azimuth angles of observation. Spline interpolation is an example of one interpolation method which can be used.

A scattered light photon number for the diffuse reflectance standard and a scattered light photon number and fluorescence photon number for the phosphor sample are determined using Formulae (9) to (11), respectively, from the data set in which photon values in dead angle directions have been interpolated.

$$\bar{W}(\lambda_{ex},\theta_i,\theta_r)=\frac{\sum_{n=0}^{N-1}W(\lambda_{ex},\theta_i,\theta_r,\phi_{r,n})}{N} \quad (9)$$

$$\bar{S}(\lambda_{ex},\theta_i,\theta_r)=\frac{\sum_{n=0}^{N-1}S(\lambda_{ex},\theta_i,\theta_r,\phi_{r,n})}{N} \quad (10)$$

$$\bar{F}(\lambda_{ex},\theta_i,\theta_r)=\frac{\sum_{n=0}^{N-1}F(\lambda_{ex},\theta_i,\theta_r,\phi_{r,n})}{N} \quad (11)$$

where

W is the scattered light photon number of the diffuse reflectance standard;

$\bar{W}$ is the averaged scattered light photon number of the diffuse reflectance standard;

S is the scattered light photon number of the phosphor sample;

$\bar{S}$ is the averaged scattered light photon number of the phosphor sample;

F is the fluorescence photon number of the phosphor sample;

$\bar{F}$ is the averaged fluorescence photon number of the phosphor sample;

λ_{ex} is the excitation wavelength;

θ_i is the angle of incidence;

θ_r is the zenith angle of observation;

$\phi_{r,n}$ is nth azimuth angle of observation;

N is the number of spectrum taken for varying azimuth angle of observation.

9.5 Luminescent radiance factor and reflected radiance factor

A luminescent radiance factor β_L or $\overline{\beta_L}$ and reflected radiance factor β_R or $\overline{\beta_R}$ at the zenith angle of observation θ_r corresponding to irradiation by excitation light at the incident angle θ_i are determined from the radiance factor of the diffuse reflectance standard $\beta_W(\lambda_{ex},\theta_i,\theta_r)$ using formulae tabulated in Table 4.

Table 4 — Formulae for calculating luminescent radiance factor and reflected radiance factor

		Luminescent radiance factor	Reflected radiance factor
Azimuth angle of observation	Fixed	$\overline{\beta_L}(\lambda_{ex},\theta_i,\theta_r)=\frac{\bar{F}(\lambda_{ex},\theta_i,\theta_r)}{\bar{W}(\lambda_{ex},\theta_i,\theta_r)}\beta_W(\lambda_{ex},\theta_i,\theta_r)$	$\overline{\beta_R}(\lambda_{ex},\theta_i,\theta_r)=\frac{\bar{S}(\lambda_{ex},\theta_i,\theta_r)}{\bar{W}(\lambda_{ex},\theta_i,\theta_r)}\beta_W(\lambda_{ex},\theta_i,\theta_r)$
	Variable	$\beta_L(\lambda_{ex},\theta_i,\theta_r)=\frac{F(\lambda_{ex},\theta_i,\theta_r)}{W(\lambda_{ex},\theta_i,\theta_r)}\beta_W(\lambda_{ex},\theta_i,\theta_r)$	$\beta_R(\lambda_{ex},\theta_i,\theta_r)=\frac{S(\lambda_{ex},\theta_i,\theta_r)}{W(\lambda_{ex},\theta_i,\theta_r)}\beta_W(\lambda_{ex},\theta_i,\theta_r)$

where

β_L is the luminescent radiance factor of the phosphor sample;

$\overline{\beta_L}$ is the averaged luminescent radiance factor of the phosphor sample;

β_R is the reflected radiance factor of the phosphor sample;

$\overline{\beta_R}$ is the averaged reflected radiance factor of the phosphor sample;

β_W is the radiance factor of the diffuse reflectance standard;

W is the scattered light photon number of the diffuse reflectance standard;

$\overline{W}$ is the averaged scattered light photon number of the diffuse reflectance standard;

S is the scattered light photon number of the phosphor sample;

$\overline{S}$ is the averaged scattered light photon number of the phosphor sample;

F is the fluorescence photon number of the phosphor sample;

$\overline{F}$ is the averaged fluorescence photon number of the phosphor sample;

λ_{ex} is the excitation wavelength;

θ_i is the angle of incidence;

θ_r is the zenith angle of observation.

9.6 Interpolation of luminescent radiance factor and reflected radiance factor at dead angle

In measurement of in-plane spatial distribution, a dead angle develops near the incident direction at values for the luminescent radiance factor and reflected radiance factor of each zenith angle of observation set for each definite angle interval. Each radiance factor in this dead angle region is estimated by interpolation. Spline interpolation is an example of one interpolation method which can be used.

9.7 External quantum efficiency

External quantum efficiency $\eta_{ext}(\lambda_{ex},\theta_i)$ is determined using Formula (12) or (13).

In the case of measurement with fixed azimuth angle of observation:

$$\eta_{ext}(\lambda_{ex},\theta_i)=\sum_{m=0}^{90^\circ/\Delta\theta_r}\beta_L(\lambda_{ex},\theta_i,m\cdot\Delta\theta_r)Z(m\cdot\Delta\theta_r) \quad (12)$$

In the case of measurement with varying azimuth angle of observation:

$$\eta_{ext}(\lambda_{ex},\theta_i)=\sum_{m=0}^{90^\circ/\Delta\theta_r}\overline{\beta_L}(\lambda_{ex},\theta_i,m\cdot\Delta\theta_r)Z(m\cdot\Delta\theta_r) \quad (13)$$

where

β_L is the luminescent radiance factor of the phosphor sample;

$\overline{\beta_L}$ is the averaged luminescent radiance factor of the phosphor sample;

λ_{ex} is the excitation wavelength;

θ_i is the angle of incidence;

θ_r is the zenith angle of observation;

$\Delta\theta_r$ is the interval of zenith angle of observation.

$Z(\theta_r)$ is a zonal coefficient, as shown in Formulae (14) and (15):

$$Z(\theta_r)=2\pi\left(1-\cos\left(\frac{\Delta\theta_r}{2}\right)\right) \quad \theta_r = 0° \tag{14}$$

$$Z(\theta_r)=4\pi\sin\left(\frac{\Delta\theta_r}{2}\right)\sin\theta_r \quad 0°< \theta_r \le 90° \tag{15}$$

where θ_r is the zenith angle of observation.

9.8 Internal quantum efficiency

Internal quantum efficiency $\eta_{int}(\lambda_{ex},\theta_i)$ is determined using Formula (16) or (17).

In the case of measurement with fixed azimuth angle of observation:

$$\eta_{int}(\lambda_{ex},\theta_i)=\frac{\sum_{m=0}^{90°/\Delta\theta_r}\beta_L(\lambda_{ex},\theta_i,m\cdot\Delta\theta_r)Z(m\cdot\Delta\theta_r)}{\sum_{m=0}^{90°/\Delta\theta_r}(1-\beta_R(\lambda_{ex},\theta_i,m\cdot\Delta\theta_r))Z(m\cdot\Delta\theta_r)} \tag{16}$$

In the case of measurement with varying azimuth angle of observation:

$$\eta_{int}(\lambda_{ex},\theta_i)=\frac{\sum_{m=0}^{90°/\Delta\theta_r}\overline{\beta_L}(\lambda_{ex},\theta_i,m\cdot\Delta\theta_r)Z(m\cdot\Delta\theta_r)}{\sum_{m=0}^{90°/\Delta\theta_r}(1-\overline{\beta_R}(\lambda_{ex},\theta_i,m\cdot\Delta\theta_r))Z(m\cdot\Delta\theta_r)} \tag{17}$$

where

β_L is the luminescent radiance factor of the phosphor sample;

$\overline{\beta_L}$ is the averaged luminescent radiance factor of the phosphor sample;

β_R is the reflected radiance factor of the phosphor sample;

$\overline{\beta_R}$ is the averaged reflected radiance factor of the phosphor sample;

Z is the zonal coefficient as described in 9.7;

λ_{ex} is the excitation wavelength;

θ_i is the angle of incidence;

$\Delta\theta_r$ is the interval of zenith angle of observation.

9.9 Absorptance

Absorptance $\alpha(\lambda_{ex},\theta_i)$ is determined using Formula (18) or (19).

In the case of measurement with fixed azimuth angle of observation:

$$\alpha(\lambda_{ex},\theta_i)=\sum_{m=0}^{90^\circ/\Delta\theta_r}\left(1-\beta_R(\lambda_{ex},\theta_i,m\cdot\Delta\theta_r)\right)Z(m\cdot\Delta\theta_r) \tag{18}$$

In the case of measurement with varying azimuth angle of observation:

$$\alpha(\lambda_{ex},\theta_i)=\sum_{m=0}^{90^\circ/\Delta\theta_r}\left(1-\overline{\beta_R}(\lambda_{ex},\theta_i,m\cdot\Delta\theta_r)\right)Z(m\cdot\Delta\theta_r) \tag{19}$$

where

β_R is the reflected radiance factor of the phosphor sample;

$\overline{\beta_R}$ is the averaged reflected radiance factor of the phosphor sample;

Z is the zonal coefficient as described in 9.7;

λ_{ex} is the excitation wavelength;

θ_i is the angle of incidence;

$\Delta\theta_r$ is the interval of zenith angle of observation.

Absorptance is calculated using Formula (20):

$$\alpha(\lambda_{ex},\theta_i)=\frac{\eta_{ext}(\lambda_{ex},\theta_i)}{\eta_{int}(\lambda_{ex},\theta_i)} \tag{20}$$

where

η_{int} is internal quantum efficiency;

η_{ext} is external quantum efficiency.

10 Test report

Report at least the following information in the test report:

a) number of this document, i.e. ISO 23946:2020;

b) date of measurement and measurement personnel;

c) sample name;

d) thickness of the sample;

e) name and model of measurement equipment;

f) radial measurement distance;

g) angle of incidence;

h) range and interval of zenith angle of observation;

i) range and interval of azimuth angle of observation for out-of-plane spatial distribution measurement;

