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Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for optical properties of ceramic phosphors for white light-emitting diodes using an integrating sphere

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Contents

Foreword

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

This second edition cancels and replaces the first edition (ISO 20351:2017), which has been technically revised.

The main changes are as follows:

- scope of the document is expanded by additionally implementing the reference material-based substitution measurement method to measure external quantum efficiency and absorptance.

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 alternatives for incandescent and fluorescent lamps. Initially, 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 were commercialised 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.

The previous edition of this document (ISO 20351:2017) was developed based on the need for standardizing a test method for internal quantum efficiency of phosphors using an integrating sphere. This standard test method has the advantage of a short measurement time and being available to those with no expertise in precise optical measurement. The external quantum efficiency and absorptance were out of the scope of ISO 20351:2017 due to an insufficient understanding of the source of variation in these measurement values.

ISO 23946 was then developed to provide alternative absolute measurement methods for the external quantum efficiency, internal quantum efficiency and absorptance using a gonio-spectrofluorometer. The application of ISO 23946 is assumed to be limited to those who intend to determine the optical properties of phosphor materials to be utilized as reference materials due to its complicated equipment and time-consuming procedure. ISO 13915 was developed following ISO 23946 and provides substitution measurement methods comparing with reference materials with values evaluated in accordance with ISO 23946 to provide external quantum efficiency, internal quantum efficiency and absorptance by using a commercially available fluorescence spectrophotometer.

It was found that the external quantum efficiency and absorptance of a phosphor material under test can also be appropriately obtained using and integrating sphere-based spectrometer with the substitution measurement method as described in ISO 13915, where the measurement using and integrating sphere-based equipment is excluded.

Therefore, the revised second edition of this document is intended to expand the scope by additionally implementing the substitution measurement method for obtaining external quantum efficiency and absorptance using an integrating sphere to the existing absolute method to obtain internal quantum efficiency. This expanded scope will benefit those who routinely use integrating sphere-based equipment.

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

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

Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for optical properties of ceramic phosphors for white light-emitting diodes using an integrating sphere

1 Scope

This document specifies test methods for the use of an integrating sphere to measure the optical properties of ceramic phosphor powders, which are used in white light-emitting diodes (LEDs) and emit visible light when excited by UV or blue light. This document specifies an absolute method to measure internal quantum efficiency and a substitution method to measure external quantum efficiency and absorptance. The substitution method uses reference materials whose external quantum efficiency and absorptance have been obtained in accordance with ISO 23946.

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 23946, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Test methods for optical properties of ceramic phosphors for white light-emitting diodes using a gonio-spectrofluorometer*

CIE S 017/E:2020, *ILV: International Lighting Vocabulary, 2nd edition*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 23946 and CIE S 017/E and the following apply.

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

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at https://www.electropedia.org/

3.1
internal quantum efficiency
ratio of the number of photons emitted in free space from the phosphor to the number of excitation light photons absorbed by the phosphor

3.2
external quantum efficiency
ratio of the number of photons emitted in free space from the phosphor to the number of excitation light photons incident on the phosphor

3.3
absorptance
ratio of the number of excitation light photons absorbed by the phosphor to the number of excitation light photons incident on the phosphor

3.4
cell
container filled with a sample or a white material such as barium sulfate

Note 1 to entry: A cell is typically a flat plate sample holder with a cylindrical hollow, a petri dish or a rectangular cell used in a spectrophotometer.

3.5
reference cell
cell filled with a white powder which has a high spectral diffuse reflectance over the whole visible spectrum (such as barium sulfate or alumina), used when measuring the excitation light spectrum

Note 1 to entry: This term is only for use in the absolute method.

3.6
white diffuser
white plate which has a high spectral diffuse reflectance over the whole visible spectrum (such as barium sulfate or polytetrafluoroethylene (PTFE)), used when measuring the excitation light spectrum

Note 1 to entry: This term is only for use in the absolute method.

3.7
secondary absorption
absorption of indirect incident light from every direction of the sphere wall by the phosphor sample

Note 1 to entry: The excitation light illuminating the sample is not entirely absorbed by the sample but is partially scattered or reflected and then repeatedly reflected on the sphere wall. Some of the scattered/reflected light can illuminate the sample again and be absorbed.

3.8
self absorption
absorption of photoluminescent photons emitted by the sample itself

4 Measurement apparatus

4.1 Apparatus configuration

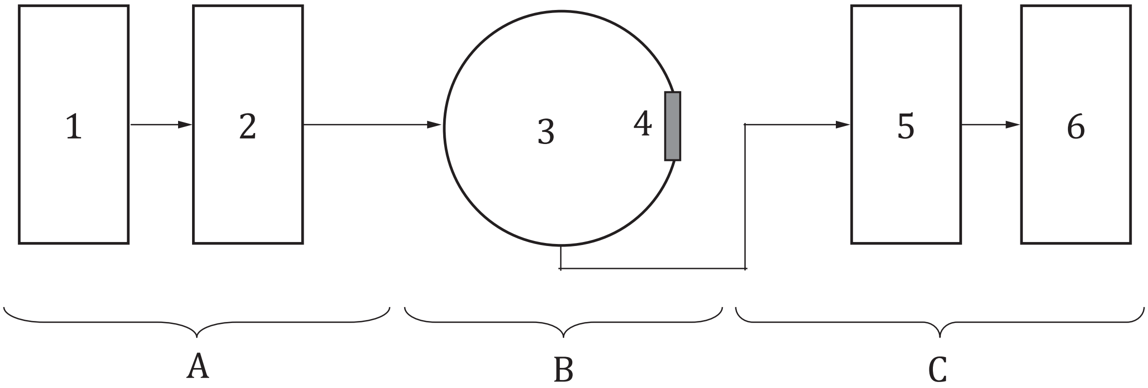
The apparatus includes a light source unit, a sample unit, a detection unit and a signal and data processing unit. Figure 1 and Figure 2 illustrate the typical configurations of a measurement apparatus.

The light source unit generates monochromatic excitation light and comprises a white light source, a power supply for the 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 and an integrating sphere.

The detecting unit comprises directing optics for collecting light, a spectrometer, a detector and an amplifier.

A fluorescence spectrophotometer equipped with a sample unit (including an integrating sphere), and equipment combining a light source unit and an array spectrometer together with the sample unit, are typical examples.

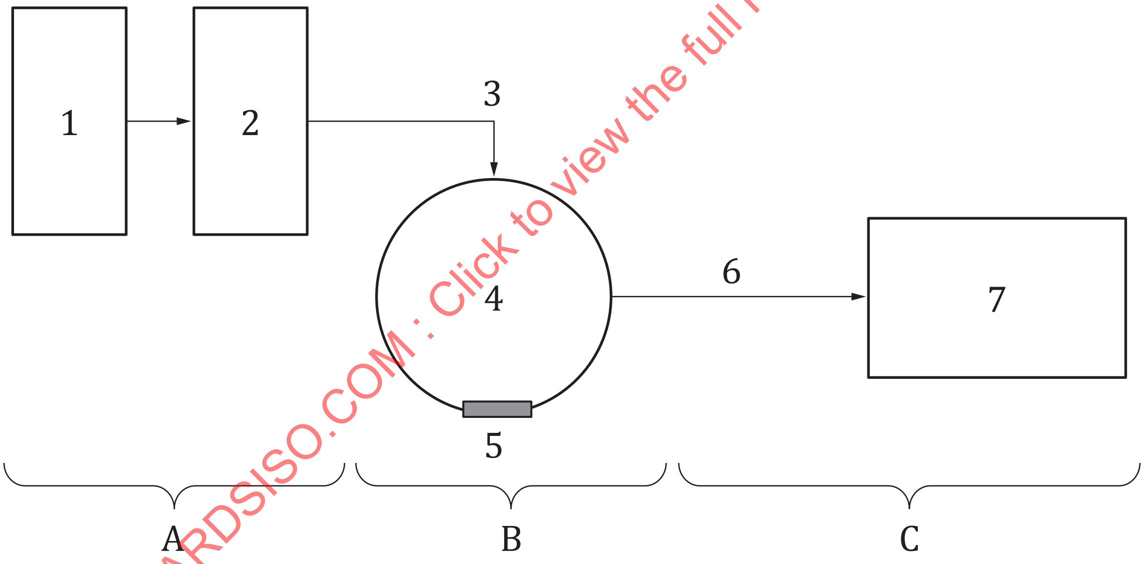


Key

A	light source unit
B	sample unit
C	detecting unit

1	light source
2	excitation monochromator
3	integrating sphere
4	cell (sample)
5	emission monochromator
6	detector

Figure 1 — Example configuration of measuring equipment (fluorescence spectrophotometer type)



Key

A	light source unit
B	sample unit
C	detecting unit

1	light source
2	monochromator
3	optical fibre
4	integrating sphere
5	cell (sample)
6	optical fibre
7	array spectrometer

Figure 2 — Example configuration of measuring equipment (array spectrometer type)

4.2 Light source unit

The spectral width of the excitation light is limited by the monochromator. The half-width of the excitation light spectrum should be 15 nm or less.

The generated excitation light is introduced into the integrating sphere via the excitation light port to illuminate a cell or a white diffuser. It is important to ensure that the beam diameter of the excitation light illuminating the sample is sufficiently smaller than the diameter of the sample facing the interior of the integrating sphere.

4.3 Sample unit

4.3.1 Cell

Ensure that the area of the sample facing the interior of the integrating sphere is sufficiently larger than that illuminated by the excitation light, and that the thickness of the sample is at least 2 mm.

When using a flat plate cell, place a cover glass on the sample. Cover with the lid when using a Petri dish.

Ensure that the cover glass, at least one side of the rectangular cell, or the Petri dish and its lid have sufficient optical transmittance over the entire measured wavelength range. Quartz is generally used for these items. Ensure that the thickness of the cover glass or the transparent side of the rectangular cell is no more than 1,25 mm.

The specification (thickness and type of material) of a cover glass or a lid shall be identical for reference material and phosphor material under test to reduce uncertainty associated with Fresnel reflection on such glass material.

Use a material with high spectral diffuse reflectance (e.g. white alumina) in the flat plate sample holder.

When the reference cell is used for absolute measurement, the sample cell should be the same type as the cell used for the reference cell. For substitution measurement, the specification of the sample cell shall be identical for reference material and phosphor material under test.

4.3.2 White diffuser or reference cell

Use a white diffuser or reference cell in the integrating sphere sample mount when measuring the excitation light spectrum.

NOTE As noted in Clause 3, a white diffuser or reference cell is only for use in the absolute method.

4.3.3 Integrating sphere

The inner wall is spherical in shape and covered with white diffused reflective material. The integrating sphere incorporates the excitation light port, the measurement port and the sample port where the cell can be mounted. For an integrating sphere with the sample port aperture, ensure that the total aperture area is less than 10 % of the total area of the integrating sphere wall. For an integrating sphere without the sample port aperture, ensure that the total aperture area in addition to the surface area of the sample is less than 10 % of the total area of the sphere wall. Use a material with sufficiently high spectral diffuse reflectance (e.g. barium sulfate, PTFE) over the entire range of measured wavelengths for the inner wall. The diameter of the integrating sphere shall be between 60 mm and 150 mm.

Note that the presence of light-absorbing material inside the sphere or on the vicinity of the sphere aperture will result in measurement errors.

Cells are placed on the inner surface of the integrating sphere, or pressed against or inserted into the sample port of the integrating sphere from the outside. The integrating sphere should be designed so that the detector is blind for the direct incidence of reflection, scattering and fluorescence from the surface of the sample.

4.4 Detection unit

4.4.1 Directing optical system

This optical component guides light collected from the measurement port of the integrating sphere into the spectrometer. The guides need to be sufficiently transparent over the entire measured spectral range. For example, a focusing lens system or an optical fibre bundle can be used.

4.4.2 Spectrometer and detector

This equipment converts light collected from the measurement port of the integrating sphere via directing optics to an electrical signal in proportion to its intensity spectrum. For example, a photomultiplier tube or a CCD detector, with its ample spectral response over the entire measured spectral range, can be used as a detector.

4.4.3 Amplifier

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

4.5 Signal and data processing unit

This unit separates and processes signals required for measurement, outputs light intensity for each measured wavelength as photon number or energy and saves the data.

5 Calibration, inspection and maintenance of measurement apparatus

5.1 General

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

5.2 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, separately verify its wavelength using a spectrometer or a wavemeter, calibrated for wavelength.

5.3 Cells and cover glasses

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

5.4 Integrating sphere walls and white diffusers

Verify that there is no contamination, damage or peeling of the walls regularly. If damage or contamination which may interfere with the high diffuse reflection factor in the range of measured wavelengths is found, clean (if possible), recycle or repaint, or replace with a new item.

5.5 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.

5.6 Spectral responsivity correction

The relative spectral responsivity of the entire measurement system from the integrating sphere to the detector should be properly calibrated in accordance with the manufacturer's instructions. All measurement spectra should be corrected based on the relative spectral responsivity calibration results.

6 Samples

6.1 Reference material

A phosphor material whose external quantum efficiency has been measured in accordance with ISO 23946 shall be used as a reference material for substitution measurement. The optical properties of the reference material should be measured under conditions as close as possible in terms of excitation wavelength and angle of incidence to those for the substitution measurement of the phosphor material under test to the reference material.

NOTE A reference material is only for use in the substitution method.

6.2 Storage and pre-processing

Phosphor samples shall be stored appropriately according to their properties and pre-processed as necessary. Samples are normally stored at room temperature in a desiccator. However, samples which react with moisture in the air, or which may be degraded with UV or visible light, shall be stored in a sealed container (filled with an inert gas using a glove box) or a coloured bottle.

Samples which readily absorb moisture shall be dried before measurement in a vacuum dry oven at a temperature at which they do not deteriorate.

6.3 Filling cells with samples

When using a flat plate cell, overfill its hollow with an excessive amount of sample, press it down with the flat plate, scrape off the excess and place the cover glass over the top. When using a rectangular cell, place the powder sample in the cell and tap it to ensure that it is densely packed, and cover with the lid if necessary. When using a Petri dish, place the powder in the dish and smooth its surface by tapping it, for example, and cover with the lid.

When the cell is mounted outside the integrating sphere, ensure that the thickness of the sample layer is sufficient to prevent leakage of excitation light. When using a Petri dish, ensure that the amount of the sample is sufficient for the results obtained to be invariant with respect to its amount.

7 Procedure, calculation and report for absolute measurement

7.1 Measurement method

7.1.1 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 the measuring apparatus on at least 30 min prior to the measurement.

7.1.2 Light spectrum without phosphor sample

Place the white diffuser or the reference cell on the sample port of the integrating sphere. When using the white diffuser, place the same cover glass as used for the sample on the diffuser. When using a Petri dish as a cell, place the dish with its lid closed on the cell mount of the integrating sphere. The cell type of the reference cell, filled with white powder as described in 6.3, should be the same as that of the phosphor sample.

Introduce the monochromated excitation light through the aperture at the excitation light port of the sphere to illuminate the white diffuser or the reference cell, and measure the intensity of the light for each wavelength at the measurement port to find the excitation light spectrum (Figure 3). The intensity of the excitation light should be controlled so as not to saturate the detector signal and deteriorate the sample, as noted in 7.1.3.

7.1.3 Light spectrum with phosphor sample

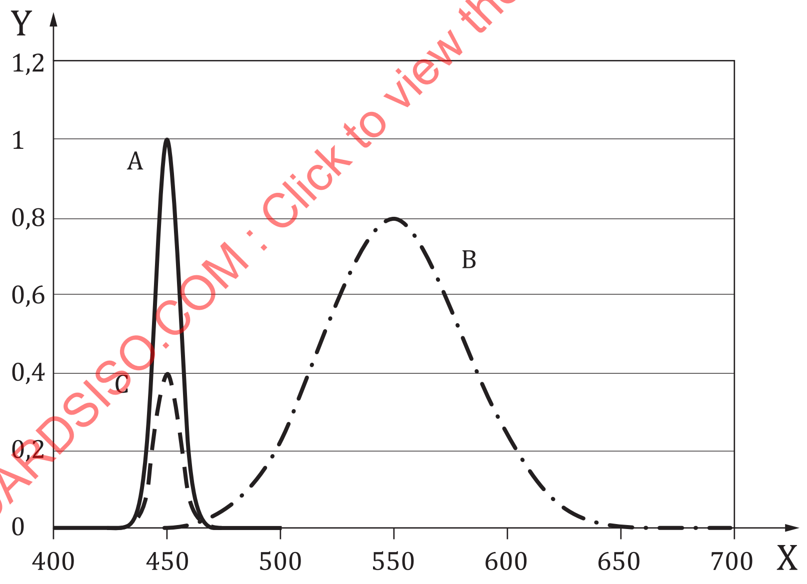
Attach the cell filled with the sample onto the sample port of the integrating sphere or mount the cell on the cell mount inside the integrating sphere.

As described in 7.1.2, illuminate the sample with excitation light then measure the light intensity with the detector to obtain the spectrum with excitation light and fluorescence components.

7.2 Calculations

7.2.1 Conversion to photon-number-based spectra

Convert the spectra measured as described in 7.1.2 and 7.1.3 to photon-number-based spectra if they are energy-based spectra.



Key

X wavelength (nm)

Y photon flux (arbitrary units)

A excitation light spectrum obtained in 7.1.2

B fluorescence spectrum obtained in 7.2.2

C excitation light spectrum obtained in 7.2.3.1

Figure 3 — Excitation light and fluorescence spectra obtained in Clause 7

7.2.2 Fluorescence spectrum

To obtain the fluorescence spectrum of the phosphor sample separately as shown in Figure 3, use the excitation light spectrum obtained in 7.1.2 to remove the excitation light spectrum component from the spectrum measured in 7.1.3. The excitation light component, for example, can be removed as follows.

a) Method 1: apply a suitable scale factor to the excitation light spectrum obtained in 7.1.2. and subtract the resulting spectrum from that obtained in 7.3 so as to obtain a smooth subtracted spectrum at the vicinity of the excitation light. Irregularities in the vicinity of the excitation wavelength, for example, can be removed by linear fitting of data points adjacently outside of the spectral area of irregularities.

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

7.2.3 Internal quantum efficiency

7.2.3.1 Relative number of absorbed photons

Extract the excitation light component (Figure 3) of the measured spectrum in 7.1.3 by subtracting the photoluminescence component extracted in 7.2.2 from the measured spectrum. Then, obtain the difference spectrum by subtracting the extracted excitation light spectrum from the measured excitation light spectrum in 7.1.2 divided by the diffuse reflectance in the excitation wavelength region of the white diffuser or reference cell, and integrate it at the spectral region of excitation light to obtain the relative number of absorbed photons for the phosphor sample.

7.2.3.2 Relative number of photoluminescent photons

Find the relative number of the photoluminescent photons emitted from the sample from the photoluminescence spectral component obtained in 7.2.2.

7.2.3.3 Internal quantum efficiency

Calculate internal quantum efficiency as follows.

$$\eta_{\mathrm{int,T}} = \frac{F}{A} \tag{1}$$

where

$\eta_{\mathrm{int,T}}$ is the internal quantum efficiency;

F is the number of photoluminescent photons;

A is the number of absorbed photons.

Corrections such as secondary absorption correction[1] and self-absorption correction[2] may be adopted, if required, in accordance with agreements between the involved personnel.

7.3 Test report

The following measurement results and information shall be included:

a) number of this document, i.e. ISO 20351:2024;

b) date of measurement and measurement personnel;

c) sample name;

d) measurement equipment;

e) wavelength of excitation light and its spectral full width at half maximum;

f) measurement spectral range;

g) internal quantum efficiency and spectral range used in calculation;

h) corrections (e.g. secondary absorption correction, self-absorption correction) used (Y/N);

i) ambient conditions;

j) any deviations from the procedure;

k) any unusual features observed.

Report the following as necessary:

l) type of light source;

m) type of detector;

n) dimensions and material of cell, thickness of sample, angle of incidence of excitation light;

o) internal diameter and material of integrating sphere;

p) material of white diffuser or powder for reference cell;

q) spectral diffuse reflectance of white diffuser or reference cell;

r) excitation light spectrum (indicate clearly whether energy-based or photon number-based);

s) measured spectrum of phosphor sample (as per 7.1.3) (as above);

t) photoluminescence spectrum (as per 7.2.2) (as above);

u) method used to remove excitation light component from measured spectrum of phosphor sample.

8 Procedure, calculation and report for substitution measurement

8.1 Measurement procedures

8.1.1 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 the measuring apparatus on at least 30 min prior to the measurement.

8.1.2 Spectrometer setup for substitution measurement

Ensure that the wavelength range for measurement be configured to cover the spectral components of scattered light and fluorescence.

When using a scanning spectrometer for the detection unit, the spectral component of scattered light at the excitation wavelength and the fluorescence spectral component are typically obtained at a single scan. In such a case, the wavelength range for measurement shall be configured to cover these spectral components. Only when these spectral components are well separated with each other, each spectral component can be measured individually.

When using an array spectrometer for the detection unit, the measurement wavelength range typically covers both spectral components of scattered light and fluorescence. When the spectral components of scattered light and fluorescence are so far apart from each other that cannot be measured within a single wavelength window, the wavelength window of the array spectrometer may be individually configured to cover each corresponding spectral component.

8.1.3 Measurement for reference material

Secure the cell filled with the reference material on the sample unit with the cell holder (if any). Verify that the dark count of the detector is sufficiently low and then begin measurement. After measurement is complete, save the spectral measurement data.

8.1.4 Measurement for phosphor material under test

Secure the cell filled with the phosphor material under test on the sample unit with the cell holder (if any). Verify that the dark count of the detector is sufficiently low and then begin measurement. After measurement is complete, save the spectral measurement data.

8.2 Calculation

8.2.1 Spectral responsivity correction

Convert each measured spectrum for reference material (see 8.1.3) and phosphor material under test (see 8.1.4), which is obtained as a data set of detection wavelength-dependent signal intensities to the corresponding spectral quantity proportional to spectral irradiance by using spectral responsivity correction data as described in 5.6.

8.2.2 Conversion to photon number-based spectral distribution

Convert each corrected energy-based spectral distribution to the corresponding photon number-based spectral distribution using Formulae (2) and (3):

$$E_{\mathrm{ph,R}}(\lambda) = E_{\mathrm{R}}(\lambda) \times \frac{\lambda}{hc} \tag{2}$$

$$E_{\mathrm{ph,T}}(\lambda) = E_{\mathrm{T}}(\lambda) \times \frac{\lambda}{hc} \tag{3}$$

where

$E_{\mathrm{ph,R}}(\lambda)$	is the photon number-based spectral intensity of the scattered light and fluorescence of the reference material;
$E_{\mathrm{ph,T}}(\lambda)$	is the photon number-based spectral intensity of the scattered light and fluorescence of the phosphor material under test;
$E_{\mathrm{R}}(\lambda)$	is the energy-based spectral intensity of the scattered light and fluorescence of the reference material;
$E_{\mathrm{T}}(\lambda)$	is the energy-based spectral intensity of the scattered light and fluorescence of the phosphor material under test;
λ	is the detection wavelength;
h	is the Planck constant;
c	is the speed of light.

8.2.3 Calculation of scattered light and fluorescence photon numbers

Calculate the scattered light photon number and fluorescence photon number based on the relative fluorescence spectrum of a phosphor sample calculated on a photon number-basis (8.2).

$$S_{\mathrm{R}} = \int_{\lambda_{\mathrm{ex}}-\Delta\lambda}^{\lambda_{\mathrm{ex}}+\Delta\lambda} E_{\mathrm{ph,R}}(\lambda)\,\mathrm{d}\lambda \qquad (4)$$

$$F_{\mathrm{R}} = \int_{\lambda_1}^{\lambda_2} E_{\mathrm{ph,R}}(\lambda)\,\mathrm{d}\lambda \qquad (5)$$

$$S_{\mathrm{T}} = \int_{\lambda_{\mathrm{ex}}-\Delta\lambda}^{\lambda_{\mathrm{ex}}+\Delta\lambda} E_{\mathrm{ph,T}}(\lambda)\,\mathrm{d}\lambda \qquad (6)$$

$$F_{\mathrm{T}} = \int_{\lambda_1}^{\lambda_2} E_{\mathrm{ph,T}}(\lambda)\,\mathrm{d}\lambda \qquad (7)$$

where

S_{R}	is the scattered light photon number of the reference material;
F_{R}	is the fluorescence photon number of the reference material;
S_{T}	is the scattered light photon number of the phosphor material under test;
F_{T}	is the fluorescence photon number of the phosphor material under test;
$E_{\mathrm{ph,R}}(\lambda)$	is the photon number-based spectral intensity of the scattered light and fluorescence of the reference material;
$E_{\mathrm{ph,T}}(\lambda)$	is the photon number-based spectral intensity of the scattered light and fluorescence of the phosphor material under test;
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 the excitation wavelength range.

If the excitation light wavelength range and the fluorescence wavelength range are adjoining, and wavelength integration according to Formulae (4–7) 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 of separation and extraction of the fluorescence component.

a) Method 1: apply a suitable scale factor to the excitation light spectrum and subtract the resulting spectrum from that obtained in 8.2 so as to obtain a smooth subtracted spectrum at the vicinity of the excitation light. Irregularities in the vicinity of the excitation wavelength, for example, can be removed by linear fitting of data points adjacently outside of the spectral area of irregularities.

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