



**International
Standard**

ISO 20351

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

Second edition

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

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document 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).

ISO draws attention to the possibility that the implementation of this document may involve the use of (a) patent(s). ISO takes no position concerning the evidence, validity or applicability of any claimed patent rights in respect thereof. As of the date of publication of this document, ISO had not received notice of (a) patent(s) which may be required to implement this document. However, implementers are cautioned that this may not represent the latest information, which may be obtained from the patent database available at www.iso.org/patents. ISO shall not be held responsible for identifying any or all such patent rights.

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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*.

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 an integrating sphere-based spectrometer with the substitution measurement method as described in ISO 13915, where the measurement using an 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

ISO/PRF 20351

<https://standards.iso.org/standards/iso/af9a3e2b-ee8d-46c8-855b-e292aa9879e2/iso-prf-20351>

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

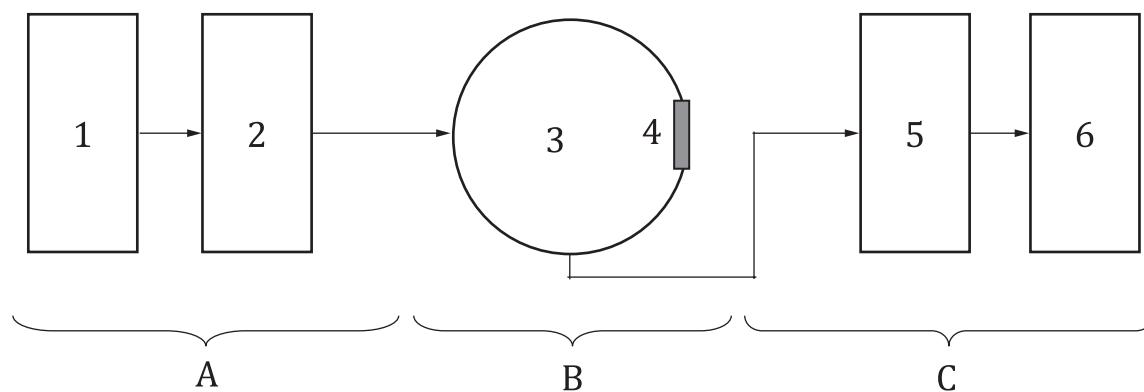
The apparatus includes a light source unit, a sample unit, a detection unit and a signal and data processing unit. [Figure 1](#) and [Figure 2](#) illustrate the typical configurations of a measurement apparatus.

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.

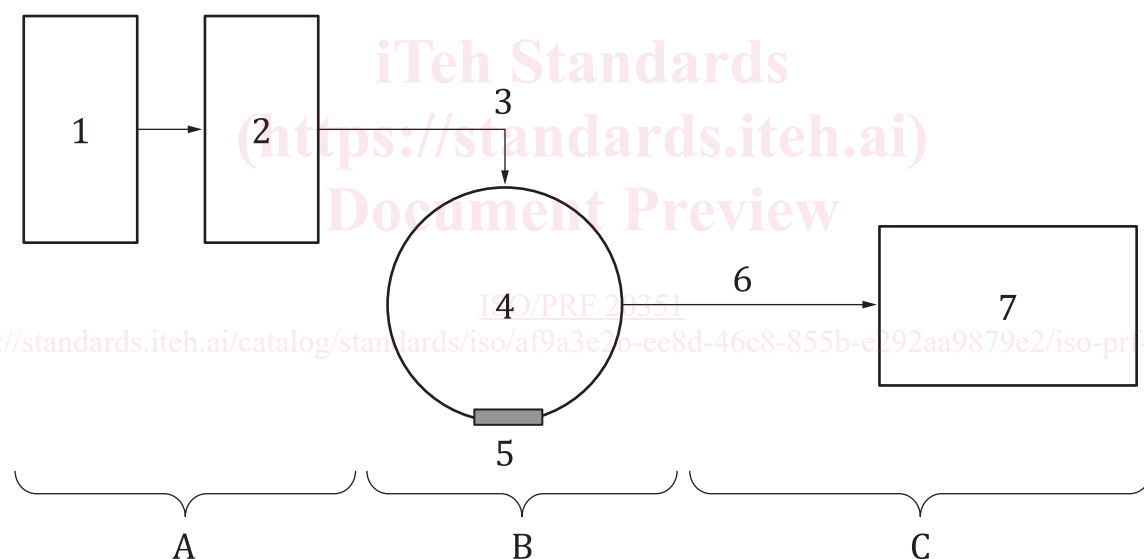


Key

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.