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Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for optical properties of ceramic phosphors for white light-emitting diodes with reference materials

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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*.

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. 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 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 a 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, the 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.

ISO 23946 was then developed to provide “integrating-sphere-free” absolute measurement methods for the external quantum efficiency, internal quantum efficiency and absorptance for ceramic phosphors for use in white LEDs using a gonio-spectrofluorometer. These goniometric measurement methods are based on basic illumination theory and can give accurate values of quantum efficiencies and absorptance for ceramic phosphors regardless of the spatial distribution of fluorescence or scattered light. While the goniometric method is theoretically rigorous, it requires large and complicated equipment as well as a long time to complete the measurement. Therefore, 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.

This document provides a simple measurement method for those who use a general-purpose instrument, where a phosphor material with optical properties evaluated according to the methods in ISO 23946 is used as a reference material.

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.

Guide to application of relevant ISO documents concerning test methods for optical properties of ceramic phosphors for white LEDs are presented in Annex C.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for optical properties of ceramic phosphors for white light-emitting diodes with reference materials

1 Scope

This document specifies a substitution measurement method to measure internal quantum efficiency, external quantum efficiency and absorptance of ceramic phosphor powders, which are used in white light-emitting diodes (LEDs) and emit visible light when excited by UV or blue light. In this method, commercially available measurement equipment, such as a fluorescence spectrophotometer or a spectroradiometer equipped with a monochromatic light source as incident light, are used to measure fluorescence spectra for reference materials whose quantum efficiencies and absorptance have been determined using the methods in ISO 23946 and a phosphor material under test.

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*

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*

ISO/CIE 11664-3, *Colorimetry — Part 3: CIE tristimulus values*

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, ISO 23946, CIE S 017/E, ISO/CIE 11664-3 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
fluorescence spectrophotometer
apparatus measuring the fluorescence spectrum of a sample irradiated on its surface by monochromatic light

4 Measurement apparatus

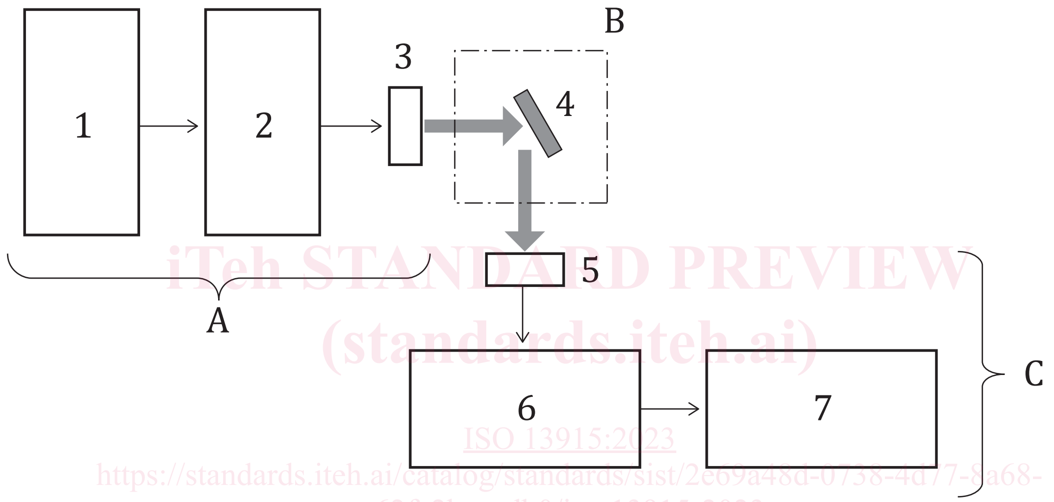
4.1 Apparatus configuration

The apparatus includes a light source unit, a sample unit, a detection unit and a signal/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 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 sample compartment and a cell holder.

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

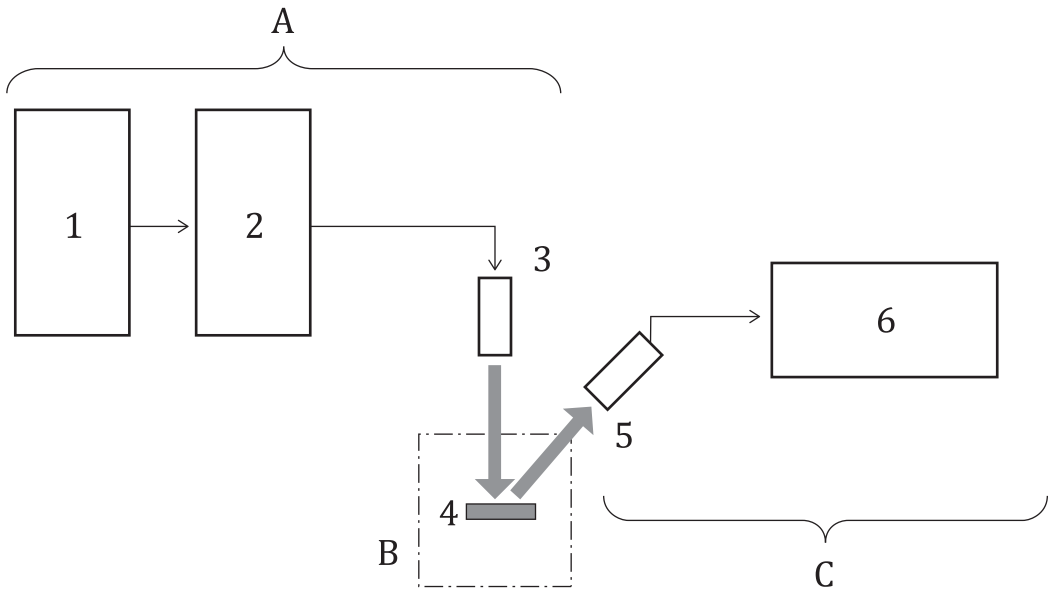


Key

A light source unit
B sample unit
C detection unit

1 light source
2 monochromator
3 optical system for irradiation
4 sample (cell)
5 directing optical system
6 spectrometer
7 detector

Figure 1 — Typical measurement apparatus configuration (fluorescence spectrophotometer type)



Key

A light source unit
B sample unit
C detection unit

1 light source
2 monochromator
3 optical system for irradiation (optical fibre probe)
4 sample (cell)
5 directing optical system (optical fibre probe)
6 array spectrometer/spectroradiometer

Figure 2 — Typical measurement apparatus configuration (array spectrometer type)

The geometrical condition of the measurement is illustrated in Figure 3. When a substitution measurement is performed with a certain fixed angle of incidence, an angle-adjustable optical system for irradiating incident light onto the centre of a sample surface may be used. The propagation vector of the optical radiation, whether emitted or reflected, is defined as the direction of observation and should be located in or near the plane of incidence.

The angle of incidence θ_i (see Figure 3) should be configured with reference to the measurement geometry applied when measuring the quantum efficiencies and absorptance of the reference material in accordance with ISO 23946.

The angle of observation θ_r (see Figure 3) shall not be identical with or close to the angle of incidence θ_i to avoid specular reflection from the surface of a cell, a cover glass or a glass lid, as well as specular-like directional scattering from the sample.

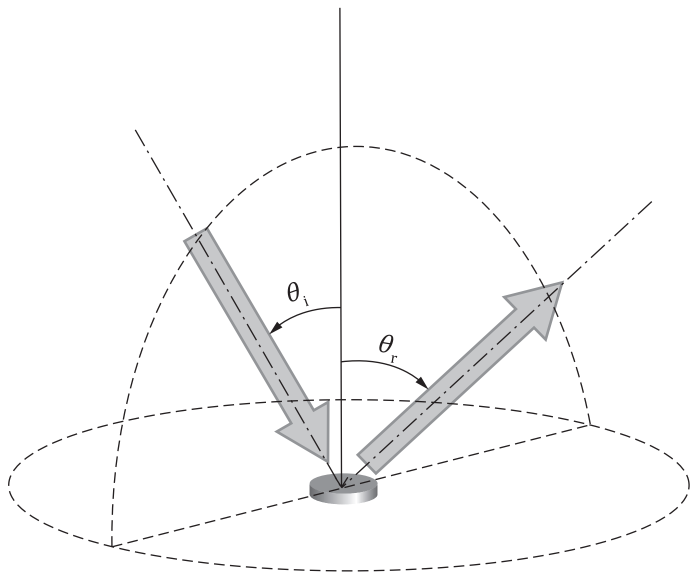
The following measurement geometries are typical configurations.

Geometry A $\theta_i = 0°$, $\theta_r = 30°$

Geometry B $\theta_i = 30°$, $\theta_r = 60°$

Geometry A is a vertical incidence configuration which is applicable to various sets of monochromatic light sources and spectroradiometers. Geometry B is the basic configuration for commercially available fluorescence spectrophotometers. Geometries other than these typical geometries are also possible.

A measurement apparatus with the sample unit comprising an integrating sphere, where the specific angle of observation cannot be defined, is out of the scope of this document. The substitution measurement can also be performed by an apparatus with an integrating sphere, which is described in ISO 20351.



Key

θ_i angle of incidence

θ_r angle of observation

Figure 3 — Geometrical condition of substitution measurement

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 excitation light passes through an optical system for irradiation and irradiates a sample. One example of an optical system for irradiation is focusing optics. The monochromated light from the exit slit of the monochromator is collimated by the focusing optics to provide a circular, nearly circular or oval-shaped beam of light onto the sample surface. An optical fibre probe can also be used as the optical system for irradiation.

The optical system for irradiation should be designed to optimise the size of the illuminated area on the sample for detecting scattered light and fluorescence efficiently.

4.3 Sample unit

4.3.1 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 incident plane shall be at least 2 mm.

A sample cell shall be made of a chemically and physically stable material which does not contaminate the sample inside and can be used in conjunction with a cell holder. A rectangular cell, a flat plate cell and a Petri dish can be used as a sample cell.

For normal incidence geometry (geometry A described in 4.1, for example), the surface of the powder sample shall be exposed directly by the excitation light: i.e., it shall not be covered with any other materials to prevent specular or diffuse reflection.

For geometries other than normal incidence (geometry B described in 4.1, for example), the surface of powder sample may be covered by a transparent plate or lid with sufficient optical transmittance over the entire measured wavelength range. The thickness and type of material of such plate or lid shall be identical for the measurement of phosphor materials under test and that of reference materials.

When using a rectangular cell, the incident side of the cell shall be transparent and have sufficient optical transmittance over the entire measured wavelength range. The rectangular cell can be placed on the cell holder so that the incident side be vertical. It may also be placed so that the incident side be horizontal only when the cell is well sealed.

When using a flat plate cell or a Petri dish, the top surface of the cell shall have a cover glass or a lid to prevent the sample powder from dispersing and contaminating its surroundings during transport or preparation for installation.

4.3.2 Sample compartment and cell holder

A sample cell can be placed inside the sample compartment. The inner surface of the compartment as well as each component incorporated inside the compartment such as a cell holder should have a matte black surface to reduce stray light. The stray light can further be reduced by appropriately placing apertures in the compartment or by giving the sample cell a slight tilt angle to block the specular reflection on the surface of the cell from entering the detector.

4.4 Detection unit

4.4.1 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. A focusing optics or an optical fibre probe can be used as a directing optical system.

4.4.2 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. A scanning monochromator is a typical example, but an array spectrometer can also be used.

4.4.3 Amplifier

This device 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 a photon number or energy and saves the associated data.

5 Calibration, inspection and maintenance of measurement apparatus

5.1 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 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.