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

Céramiques fines (céramiques avancées, céramiques techniques avancées) — Mesurage absolu du rendement quantique interne des luminophores des diodes électroluminescentes blanches en utilisant une sphère intégrante

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Foreword

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

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. 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 capitalizing 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 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 the optical properties of the phosphors in a well-established manner. However, standard measurement methods of studying the optical properties of luminescent powder materials commercially used for white LEDs have never been developed.

Photoluminescence quantum efficiency is one of the key parameters of phosphors for use in white LEDs and has been measured extensively by using an integrating sphere-based absolute method. This method was originally developed to determine the photoluminescence quantum efficiency for fluorophore-doped organic thin films and solutions, and has also been applied to phosphor powders. However, those who measure the quantum efficiency of phosphor materials have frequently noted that the measured quantum efficiency may deviate beyond their tolerance level, depending on the measurement equipment, the geometrical configuration of the integrating sphere and the arrangement of the sample cell, even if the measurement procedure is common in principle. This document provides the absolute measurement method of internal quantum efficiency of phosphors for use in white LEDs with reduced deviation of measured values. In this document, measurement equipment and procedures, which can be the sources of the deviation, are described in detail, helping those who address the high performance phosphors for competitive SSL products to obtain the proper information on their competitiveness.

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1 Scope

This document specifies a method of absolute measurement (using an integrating sphere) of internal quantum efficiency of phosphor powders which are excited by UV or blue light and emit visible light, and which are used for white light-emitting diodes (LEDs).

This document can be adopted for the measurement of phosphors used in non-white LEDs, for example, green, orange, pink or purple LEDs.

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.

CIE S 017/E:2011, *International Lighting Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in CIE S 017/E:2011 and the following apply.

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

— ISO Online browsing platform: available at http://www.iso.org/obp

— IEC Electropedia: available at http://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
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.

3.3
reference cell
cell (3.2) 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

3.4
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

3.5
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.

3.6
self absorption
absorption of photoluminescent photons emitted by the sample itself

4 Measuring equipment

4.1 Equipment configuration

The equipment comprises a light source unit, a sample unit, a detecting unit, and a signal and data processing unit.

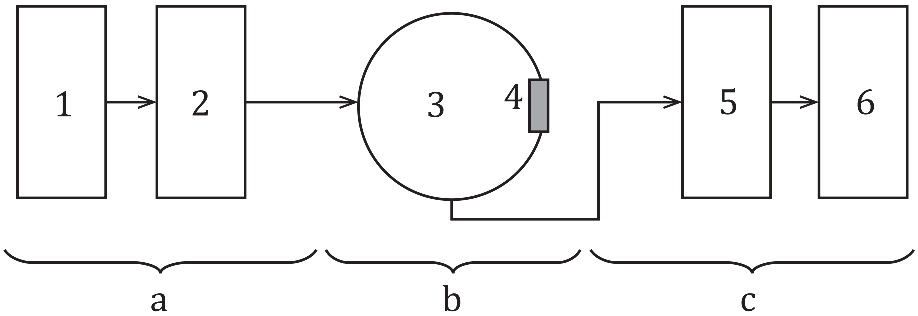
The light source unit generates excitation light and comprises a white light source, a power supply for the light source, a focusing optics and a wavelength selection unit (monochromator for the light source). A laser can also be used as the light source.

The sample unit comprises a cell and an integrating sphere.

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

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

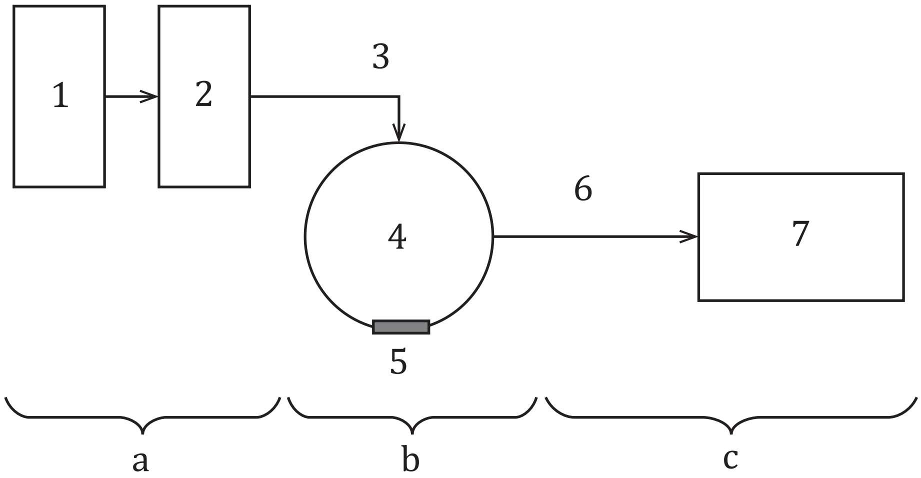
Typical configurations for measuring equipment are shown in Figure 1 and Figure 2.



Key

1 light source
2 excitation monochromator
3 integrating sphere
4 cell (sample)
5 emission monochromator
6 detector
a light source unit
b sample unit
c detecting unit

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



Key

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

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. It is desirable that the half-width of the excitation light spectrum is less than 15 nm.

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 sample holder, 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.

Use a material with high spectral diffuse reflectance (e.g. white alumina) in the flat plate sample holder. When the reference cell is used, the sample cell should be the same type as the cell used for the reference cell.