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

iTeh STANDARD PREVIEW
(standards.iteh.ai)

[ISO 23946:2020](https://standards.iteh.ai/catalog/standards/sist/e7e65a0e-10bd-4d42-9d4f-a8ebaac4d8c2/iso-23946-2020)

<https://standards.iteh.ai/catalog/standards/sist/e7e65a0e-10bd-4d42-9d4f-a8ebaac4d8c2/iso-23946-2020>



iTeh STANDARD PREVIEW (standards.iteh.ai)

ISO 23946:2020

<https://standards.iteh.ai/catalog/standards/sist/e7e65a0e-10bd-4d42-9d4f-a8ebaac4d8c2/iso-23946-2020>



COPYRIGHT PROTECTED DOCUMENT

© ISO 2020

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

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

Published in Switzerland

Contents

	Page
Foreword	v
Introduction	vi
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Spherical coordinate system	1
5 Measurement apparatus	2
5.1 Apparatus configuration	2
5.2 Light source unit	4
5.3 Sample unit	4
5.3.1 Cell	4
5.3.2 Diffuse reflectance standard	4
5.3.3 Sample stage	4
5.4 Detection unit	5
5.4.1 Directing optical system	5
5.4.2 Spectrometer and detector	5
5.4.3 Amplifier	5
5.5 Rotational positioning unit	5
5.5.1 Mechanism for setting angle of incidence	5
5.5.2 Mechanism for setting zenith angle of observation	5
5.5.3 Mechanism for setting azimuth angle of observation	5
5.6 Enclosure	5
5.7 Signal and data processing unit	6
6 Calibration, inspection and maintenance of measurement apparatus	6
6.1 General	6
6.2 Wavelength calibration of light source unit	6
6.3 Cells	6
6.4 Diffuse reflectance standard	6
6.5 Wavelength calibration of detection unit	6
6.6 Spectral responsivity calibration	6
6.7 Rotational positioning unit	6
7 Samples	6
7.1 Storage and pre-processing	6
7.2 Filling cells with samples	7
8 Measurement procedures	7
8.1 Measurement environment	7
8.2 In-plane spatial distribution measurement	7
8.2.1 Goniometric measurement of diffuse reflectance standard	7
8.2.2 Gonio-spectrofluorometric measurement of phosphor sample	7
8.3 Measurement of spatial light distribution with varying azimuth angle of observation	8
8.4 Evaluation of surface uniformity with varying azimuth rotational angle of sample	8
9 Calculation	8
9.1 Relative spectral distribution	8
9.1.1 Spectral responsivity and accumulation time corrections	8
9.1.2 Mean spectrum for varying azimuth rotational angle of sample	8
9.2 Conversion to photon number-based spectral distribution	9
9.3 Calculation of scattered light and fluorescence photon numbers	10
9.3.1 Scattered light photon number for diffuse reflectance standard	10
9.3.2 Scattered and fluorescence photon numbers for phosphor sample	11
9.4 Average of scattered light or fluorescence photon number for variable azimuth angle of observation	12

9.5	Luminescent radiance factor and reflected radiance factor	13
9.6	Interpolation of luminescent radiance factor and reflected radiance factor at dead angle	14
9.7	External quantum efficiency	14
9.8	Internal quantum efficiency	15
9.9	Absorptance	15
10	Test report	16
Annex A (informative) Gonio-spectrofluorometric measurement for less absorptive samples		18
Bibliography		20

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 23946:2020

<https://standards.iteh.ai/catalog/standards/sist/e7e65a0e-10bd-4d42-9d4f-a8ebaac4d8c2/iso-23946-2020>

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 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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 $\theta_i = \theta_r = 0^\circ$, and the plane of incidence is defined as $\phi_r = 0^\circ$.

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.

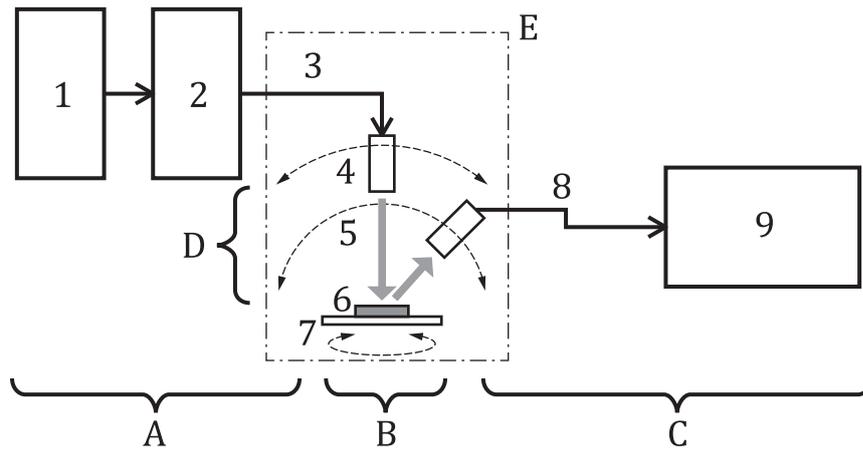
The light source unit generates monochromatic excitation light and comprises a white light source, a power supply for the white light source, a focusing optical system, a wavelength selection unit (monochromator for the white light source) and an optical system for irradiation. A collimated laser beam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

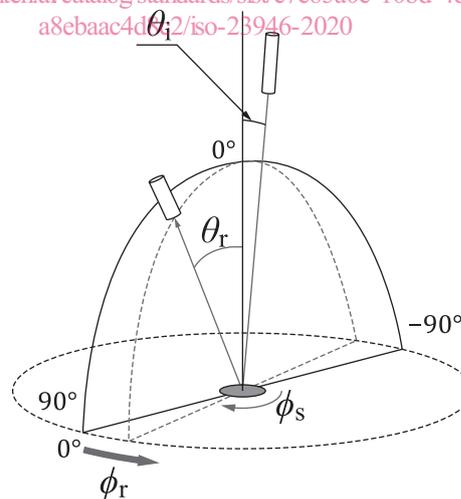


Key

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

<https://standards.itech.ai/catalog/standards/sist/e7e65a0e-10bd-4d42-9d4f-a8ebaac4d012/iso-23946-2020>



Key

θ_r	zenith angle of observation	ϕ_r	azimuth angle of observation
θ_i	angle of incidence	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.

<https://standards.iteh.ai/catalog/standards/sist/e7e65a0e-10bd-4d42-9d4f-a8ebaac4d8c2/iso-23946-2020>

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.