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INTERNATIONAL STANDARD

NORME INTERNATIONALE

colour
inside

Nanomanufacturing – Key control characteristics
Part 3-1: Luminescent nanomaterials – Quantum efficiency

Nanofabrication – Caractéristiques de contrôle clé
Partie 3-1: Nanomatériaux luminescents – Rendement quantique

IEC 62607-3-1:2014-05(en-fr)

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NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS

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FOREWORD

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The text of this standard is based on the following documents:

FDIS	Report on voting
113/214/FDIS	113/219/RVD

Full information on the voting for the approval of this standard can be found in the report on voting indicated in the above table.

This publication has been drafted in accordance with the ISO/IEC Directives, Part 2.

A list of all parts of the IEC 625607 series, published under the general title *Nanomanufacturing – Key control characteristics,* can be found on the IEC website.

The committee has decided that the contents of this publication will remain unchanged until the stability date indicated on the IEC web site under "http://webstore.iec.ch" in the data related to the specific publication. At this date, the publication will be

- reconfirmed,
- withdrawn,
- replaced by a revised edition, or
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INTRODUCTION

One of the principal drivers of solid-state lighting (SSL) is the potential efficiency of the illumination devices to convert electricity into light. Incandescent and fluorescent lighting devices are only about 5 % to 30 % efficient, with incandescent lighting having the lowest efficiency. Since a significant portion of all electricity consumed is used in providing lighting, increasing the efficiency of lighting devices will have a huge impact on the world’s energy consumption. The luminous efficiency of SSL devices is a critical measurement of their overall efficiency, and standard methods to perform these measurements have been established and were essential to producing reliable product information for manufacturers and consumers. The same is true of the luminescent materials on which these light-emitting diode (LED) manufacturers rely; however, no such standard currently exists. This standard provides SSL manufacturers a universal means for comparing luminescent nanomaterials from different suppliers, and potentially for luminescent materials for LEDs in general.

The most common SSL devices are composed of a blue light-emitting diode (LED) and a luminescent material. The blue LED optically excites the luminophore, which will radiate light of the appropriate colour or colours to yield the desired white spectrum. This device, termed a phosphor-converted light emitting diode (or pc-LED), converts the electricity indirectly into white light by first creating blue light and then converting the blue light into broad-band visible radiation. Currently, quantum dots (QDs) or nanophosphors are one option for the photoluminescent material that converts the blue LED wavelength to broad spectrum visible light. QDs and nanophosphors are of interest in this application for several reasons including their greater colour flexibility, narrowband emission spectrum, broadband absorption, near-infinite flocculation time, reduced bleaching, and lower scattering compared to conventional phosphors which are typically larger than 5 μm. QD-enabled pc-LEDs have been shown to have the best possible combination of colour rendering, correlated colour temperature, and luminous efficiency of any other pc-LED on the market.

A critical measurement parameter for luminescent materials used in the lighting industry is quantum efficiency, which is defined in this standard as the number of photons emitted into free space by a luminescent nanoparticle divided by the number of photons absorbed by the nanoparticle. Suppliers of QDs and luminescent nanomaterials typically measure only relative quantum efficiency (or alternatively, quantum yield) in the solution phase due to the ease of such measurements and the applicability of such measurements to biomedical imaging (a widespread use of QDs in R&D). These measurements are often taken at low concentrations where effects such as nanoparticle agglomeration and re-absorption are minimized. However, in end-use applications, the actual concentration of luminescent nanomaterials may be significantly different. For example, concentrated luminescent nanoparticle formulations (in either solid or liquid state) may be required to achieve a desired luminous flux and correlated colour temperature in a SSL device. This standard codifies that method for the first time, and establishes an absolute quantum efficiency test method for both solid (e.g., luminescent nanoparticles embedded in polymer matrices, coated on glass optics, applied directly to light emitted diodes, and other form factors) and solution samples (e.g., colloidal suspensions of luminescent nanoparticles), enabling suppliers and purchasers to compare the performance of one material to another, both in their raw (solution) phase as well as their technologically relevant (solid) phase of matter.

NANOMANUFACTURING –
KEY CONTROL CHARACTERISTICS

Part 3-1: Luminescent nanomaterials –
Quantum efficiency

1 Scope

This part of IEC 62607 describes the procedures to be followed and precautions to be observed when performing reproducible measurements of the quantum efficiency of luminescent nanomaterials. Luminescent nanomaterials covered by this method include nano-objects such as quantum dots, nanophosphors, nanoparticles, nanofibers, nanocrystals, nanoplates, and structures containing these materials. The nanomaterials may be dispersed in either a liquid state (e.g., colloidal dispersion of quantum dots) or solid-state (e.g., nanofibers containing luminescent nanoparticles). This standard covers both relative measurements of liquid state luminescent nanomaterials and absolute measurements of both solid and liquid state nanomaterials.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

3 Terms and definitions

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

NOTE See also ISO TS 80004-2 (in preparation).

3.1
absorbance

negative base 10 logarithm of the ratio of the intensity of light (I) that has passed through and transmitted by a sample to the incident intensity (I_0) at a specified wavelength

Note 1 to entry: Expressed mathematically, absorbance = $-\log(I/I_0)$. Proper corrections are required for other losses (e.g., reflection and scattering) for this equation to be correct.

3.2
absorptance

ratio of the radiant or luminous flux in a given spectral interval that is absorbed by a medium to that of the incident light source

Note 1 to entry: The sum of the hemispherical reflectance, the hemispherical transmittance, and the absorptance is one.

3.3
absorption

process by which matter removes photons from incident light and converts it to another form of energy such as heat

Note 1 to entry: All of the incident photon flux is accounted for by the processes of absorption, reflection, and transmission.

3.4
collimated incident light method

method of determining absolute quantum efficiency that utilizes a collimated light beam, such as a laser, which is introduced into an integrating sphere containing the sample to be measured

3.5
diffuse incident light method

a method of determining absolute quantum efficiency that utilizes a diffuse light beam from a laser, light emitting diode or other source, which is introduced into an integrating sphere containing the sample to be measured

3.6
matrix

components of a sample other than the material being analyzed

Note 1 to entry: Matrix materials are typically inert organic or inorganic materials that contain luminescent nanoparticles.

3.7
nanomaterial

classification of materials that encompasses both nano-objects and nanostructured materials

Note 1 to entry: Nano-objects are materials with one, two, or three dimensions in the size range from 1 to 100 nanometres.

3.8
optical density
OD

negative base 10 logarithm of the ratio of the intensity of light that has passed through a sample, at a specified wavelength, to the intensity of the incident light source at that wavelength

Note 1 to entry: The abbreviation for optical density is OD. The optical density and absorbance of a sample are the same, if reflection losses have first been taken into account.

3.9
photobleaching

phenomenon occurring in luminescent nanomaterials in which the fluorescent characteristic of the nanomaterial is degraded or destroyed by the light exposure necessary to initiate photoluminescence

Note 1 to entry: The net result of photobleaching is a decrease in quantum efficiency over time.

3.10
photobrightening

phenomenon occurring in quantum dots and other luminescent nanomaterials in which the intensity of light emission from the nanomaterials, at a constant incidence flux, gradually increases over a period of time

Note 1 to entry: The net result of photobrightening is an increase in quantum efficiency over time.

3.11
power conversion efficiency

ratio of the optical power in the emitted radiation divided by the optical power required to produce the radiation

3.12
quantum dot
semiconductor nanocrystal that exhibits size dependent properties due to quantum confinement effects on the electronic states

3.13
quantum efficiency
efficiency of photon emission from luminescent nanoparticles

Note 1 to entry: Quantum efficiency is also known as quantum yield.

Note 2 to entry: Quantum efficiency for luminescent nanomaterials is the ratio of the number of emitted photons to the number of absorbed photons. For the purposes of this standard, the measured quantum efficiency is a measure of the photons radiated by the luminescent nanomaterials into free space, and is more a measure of external quantum efficiency.

3.14
relative quantum efficiency
quantum efficiency measured relative to that of a well-characterized standard reference material

3.15
absolute quantum efficiency
quantum efficiency determined by measuring a value directly proportional to the number of photons emitted and absorbed

Note 1 to entry: The calibration standards used to determine absolute quantum efficiency shall be traceable to primary standards or national reference standards (e.g. NIST).

3.16
external quantum efficiency
ratio of the total number of photons emitted into free space by a luminescent material to the number of photons absorbed by the material

Note 1 to entry: For the purposes of this standard, external quantum efficiency (EQE) and quantum efficiency are used interchangeability.

3.17
internal quantum efficiency
ratio of the total number of photons emitted by a luminescent material internal to a device or material, to the number of photons absorbed by the material, regardless of whether the photons are emitted into free space

Note 1 to entry: The distinction between internal quantum efficiency (IQE) and external quantum efficiency (EQE) is that IQE includes all photons emitted by a luminescent material whereas EQE includes only those photons emitted into free space.

3.18
radiant energy
Q
energy travelling as electromagnetic waves

Note 1 to entry: Radiant energy is usually expressed in joules or watts times seconds. A quantum of radiant energy is a photon.

3.19
radiant flux
Φ
time rate flow of radiant energy

Note 1 to entry: Radiant flux is typically expressed in watts

3.20
spectral radiant flux
radiant flux per unit wavelength interval at a given wavelength (λ)

Note 1 to entry: Spectral radiant flux is typically denoted by Φ_λ, which is equivalent to $d\Phi/d\lambda$, and is usually expressed in units of watts per nm.

3.21
standard reference material
SRM
material which has been characterized to be sufficiently homogeneous and stable with respect to one or more specified properties

Note 1 to entry: SRMs are accompanied by a certificate which certifies the values of these properties that have been established with traceability to the accurate realization of the unit and each certified value includes a stated uncertainty with a given level of confidence (see also SIPM Metrology brochure, 2nd edition, December 2003).

4 General notes on tests

4.1 General

It is recommended that good laboratory practices be exercised in conducting measurements on the quantum efficiency of luminescent nanomaterials as described in this document. In particular, the area where measurements are taken should be clean and free of dirt and debris and their sources.

4.2 Ambient onditions

Test equipment shall be located in an area with stable ambient (25 ± 2) °C, relative humidity, and consistent air flow. Locations underneath heating, ventilation, or air conditioning vents or by large fans shall be avoided since the change in air movement may adversely impact measurements. Ambient room temperature shall be measured in a consistent manner and reported with test results. When measuring room temperature, the temperature sensor shall be shielded from direct optical radiation from any source.

In addition, since stray light could influence the measurement results, background lighting should be held to the lowest possible level during all measurements.

4.3 Photobrightening and photobleaching

When irradiated with high intensity excitation sources, luminescent nanoparticles can exhibit both photobrightening (where the emission efficiency of the material increases during irradiation) and photobleaching (where the emission efficiency decreases during irradiation). Photobrightening can be either reversible (whereby the efficiency equilibrates to the original value once the excitation source is removed from the sample) or irreversible. Photobleaching is often irreversible due to physical damage or degradation of the material. These two phenomena can lead to erroneous efficiency measurements and therefore, care should be taken to eliminate or reduce their effects during measurement by closely monitoring the light exposure history of the sample. Consideration should be given to the excitation power applied to the sample (should be minimized while not sacrificing signal-to-noise) as well as the exposure time the excitation is applied to the sample (should be kept to a minimum while not sacrificing signal-to-noise).

4.4 Luminescence from contaminants at Illumination wavelengths < 380 nm

Airborne contaminants such as smoke, hydrocarbons and fabric lint can accumulate in an integrating sphere over time. These contaminants can fluoresce under UV irradiation (< 380 nm) and hence cause attenuation of excitation signal and/or emission signal. In addition, some highly reflecting materials used for coating the integrating spheres may exhibit intrinsic parasitic emissions that cannot be removed by cleaning the sphere. The fluorescence effect is amplified because of multiple reflections inside the integrating sphere. Therefore, it is important to characterize the fluorescence properties of the integrating sphere and make suitable corrections particularly while conducting measurements with UV excitation sources. Procedures are available for correcting for this stray luminescence [1,2][1].

4.5 Industrial hygiene

Limited information is presently available on the environmental, health, and safety effects of nanomaterials in general. As such, the effects of human exposure to nanomaterials are unknown, however international exposure standards are under development. Prudent laboratory methods should be followed to minimize exposure to nanomaterials until additional information is available. Information and recommendations on the safe handling of nanomaterials are available and should be consulted.

NOTE One reference is U.S. National Institute for Occupational Safety and Health publication 2009-125 [3].

5 Measurement of relative quantum efficiency of nanomaterials

5.1 General

Relative measurements of quantum efficiency are performed using a standard reference material with well-characterized properties. Due to the widespread use of relative measurement methods, there are a number of references that describe the instrumentation and setup procedures for fluorescence measurements [4, 5, 6, 7]. An example of a standard reference material used in relative quantum efficiency measurements is the use of a fluorescent organic dye of known quantum efficiency in determining the quantum efficiency of a colloidal suspension of quantum dots. Examples of the quantum efficiency of some typical standards can be found elsewhere [8]. The initial step of this procedure is to prepare a calibration curve over a specific spectral region using the fluorescent organic dye. The quantum efficiency of a sample is then determined relative to this calibration curve. Measurements of this type are typically performed on liquid-phase materials, as the fluorescent dye standards may be readily produced as liquid solutions of known concentrations.

5.2 Test equipment

5.2.1 Required supplies and test equipment

Test equipment for relative measurements of quantum efficiency shall include the following:

- standard fluorescence quartz cuvette of known path length. In the discussion below, it is assumed that cuvettes with a path length of 10 mm are used. If different sizes of cuvettes are used, appropriate adjustments in solution volumes may be necessary;

  NOTE Incomplete cleaning of the cuvettes may leave residues that could negatively impact quantum efficiency measurements. It is good practice to acid-wash all quartz cuvettes before use to ensure that all residual quantum dots are removed from the cuvette prior to measurements.

- microbalance;
- microsyringe;
- spectrophotometer with diffuse transmittance capability that measures absorption over the spectral region of interest (typically the ultraviolet and visible (UV-Vis) regions). Wavelength calibration of the spectrophotometer should be verified at least annually using a light source of well-characterized emission wavelengths, such as a mercury argon calibration source;
- fluorescence spectrophotometer capable of producing excitation radiation in the spectral region of interest (typically UV-Vis) and measuring the excitation and emitted radiation. Additional information on the setup and calibration of fluorescence spectrophotometers

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[1] Numbers in square brackets refer to the Bibliography.

can be found elsewhere [4, 5, 6, 7,9,13]. Typically the excitation radiation is produced by a monochromated discharge lamp with an adjustable slit at the exit of the monochromator to control peak full-width-at-half-maximum (FWHM). Emitted radiation from the sample typically passes through additional optics including an emission slit and monochromator, and then strikes the detector (e.g., photomultiplier tube). A calibration file for the spectral response of the emission monochromator and detector is needed and should either be obtained from the instrument manufacturer or created from a calibrated light source.

5.2.2 Test equipment setup

5.2.2.1 UV-Vis spectrophotometer

The UV-Vis spectrophotometer shall be set to scan the spectral region of interest, which is typically set from 300 nm to 800 nm. Acquisition parameters for the spectrophotometer shall be adjusted to achieve an optimal signal-to-noise ratio. For example, the minimum absorbance shall be set at -0,05 and the maximum absorbance set at 1,00.

5.2.2.2 Fluorescence spectrophotometer

In measuring sample fluorescence with a fluorescence spectrophotometer, it is necessary to specify an excitation wavelength and wavelengths to start and end collection of the emission spectra. In choosing these wavelengths, consideration should be given to minimize the overlap region between the red edge of the excitation spectrum and blue edge of the emission spectrum where re-absorption of the fluorescence occurs. Evidence has shown that the OD in the overlap region is typically less than 0,05 to minimize re-absorption or inner-filter effects. In addition, the slit width on both the excitation and emission monochromators shall normally be set to the same value (see Table 1). In determining the slit width, there is a trade-off between signal intensity and peak resolution. It is recommended that the slit width be set to the minimum value that does not adversely affect signal-to-noise ratio. However, the spectral bandpass conditions (i.e., slit width multiplied by the reciprocal linear dispersion of the monochromator) shall remain invariant for the measurement of the sample and reference material. Other spectrophotometer properties such as photomultiplier tube (PMT) voltage shall also be the same for sample and reference materials.

Since fluorescence measurements are typically carried out over a narrower spectral region than absorbance measurements, different pre-determined acquisition protocols can be programmed into many instruments. Representative values from three methods, “Green QY method”, “Red QY method – high QY”, and “Red QY method – low QY” are provided in Table 1. In this example, QY stands for quantum yield. Different methods (and associated fluorescent material standards) would be required for other spectral regions of interest.

Table 1 – Example fluorescence methods for relative measurements

	Green QY method	Red QY method – high QY	Red QY method – low QY
Excitation	465 nm	540 nm	530 nm
Start collection	470 nm	545 nm	540 nm
End collection	700 nm	800 nm	850 nm
Excitation slit	2,5 nm	2,5 nm	2,5 nm
Emission slit	2,5 nm	2,5 nm	2,5 nm
PMT detector voltage	Medium	Medium	High

5.3 Calibration

5.3.1 General

Fluorescent materials with well characterized quantum efficiencies shall be used as calibration standards for relative measurements of solutions. In choosing a reference material, it is important that the excitation wavelength of the reference material be similar to the expected excitation wavelength of the sample in the intended application. It is also important that the quantum efficiency of the reference material used in relative quantum efficiency measurements be equal to or greater than the expected value for the samples undergoing test. For SSL applications, the excitation wavelength often resides between 440 nm and 470 nm, although other excitation wavelengths may be used. The emission wavelength of the standard should also be similar to that of the luminescent nanomaterials sample. A list of potential reference materials may be found in reference [8] and other sources. Depending upon the spectral region of interest, multiple fluorescent materials may be required to provide accurate calibration. Examples of possible quantum efficiency calibration standards for relative measurements are provided in Table 2. Calibration standards for other wavelengths of interest can be found in reference [8].

Table 2 – Suggested calibration standards for relative quantum efficiency measurements of luminescent nanoparticle solutions

Fluorescent standard	Solvent	Excitation wavelength (nm)	Emission wavelength range (nm)	Method used	Quantum efficiency	Reference
Rhodamine 560	Ethanol	465	470 – 700	Green	0,92	10
Cresyl Violet	Methanol	540	540 – 850	Red	0,54	8,11
Rhodamine 101	Ethanol	465, 540	450 – 750	Green, red	1,00	8,12

5.3.2 Calibration standard – preparation

5.3.2.1 Concentrated stock solution

Using a microbalance, weigh out approximately 2 mg of the fluorescent calibration standard in a 20 ml vial. Dissolve the dye in 10 ml of the appropriate solvent to create a concentrated stock solution of the calibration standard.

5.3.2.2 Dilute stock solution

Remove 2 ml of the concentrated stock solution via syringe and place it into a 20 ml vial. Dilute the solution with an additional 8 ml of original solvent to create the dilute stock solution of the calibration standard.

5.3.3 Calibration standard – test measurements

5.3.3.1 Initial measurement

Remove 2,5 ml of solvent and place into a quartz cuvette. Then run a baseline in the UV-Vis spectrophotometer.

Using a microsyringe, add 100 µL of the calibration standard dilute stock solution to the cuvette and mix well.

Take an absorbance measurement on the UV-Vis spectrophotometer and note the optical density (OD) at the excitation wavelength of choice.

5.3.3.2 Maximum concentration measurements

Divide the noted OD by 100 in order to calculate an OD/µL stock solution ratio. With this number, concentrate or dilute the quartz cuvette solution to the point where the OD at the excitation wavelength is 0,05 (see Figure 1).

Check this calculation by taking an absorbance measurement on the spectrophotometer.