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TECHNICAL SPECIFICATION

Nanomanufacturing – Material specifications –
Part 4-1: Luminescent nanomaterials – Blank detail specification

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NANOMANUFACTURING – MATERIAL SPECIFICATIONS –

Part 4-1: Luminescent nanomaterials – Blank detail specification

FOREWORD

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- the subject is still under technical development or where, for any other reason, there is the future but no immediate possibility of an agreement on an International Standard.

Technical Specifications are subject to review within three years of publication to decide whether they can be transformed into International Standards.

IEC TS 62565-4-1, which is a Technical Specification, has been prepared by IEC technical committee 113: Nanotechnology for electrotechnical products and systems.

The text of this Technical Specification is based on the following documents:

Enquiry draft	Report on voting
113/476/DTS	113/508/RVDTS

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

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

A list of all parts in the IEC 62565 series, published under the general title *Nanomanufacturing – Material specifications*, can be found on the IEC website.

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

- reconfirmed,
- withdrawn,
- replaced by a revised edition, or
- amended.

A bilingual version of this publication may be issued at a later date.

INTRODUCTION

Lighting devices and displays are transitioning from incandescent illumination sources based on heated filaments to solid-state lighting (SSL) sources. In devices such as lamps and luminaires used for general illumination, light emitting diodes (LED) form SSL sources that provide light, and a wide variety of lighting colours are commercially available. In display products such as liquid crystal devices, white backlights are used in conjunction with colour filters to provide red, green and blue colours, and these backlights are also increasingly leveraging breakthroughs in LED technologies to increase the colour gamut. There are several key drivers for this change including increased energy efficiency, increased product lifetime, flexibility in colours produced and good colour rendering properties. For example, solid-state lighting (SSL) sources can achieve luminous efficacies that are significantly higher than conventional incandescent lamps. Since approximately 20 % of the world's electricity consumption is attributed to providing illumination, the impact of such a large gain in luminous efficacy provided by changing to SSL technologies is significant. Likewise, SSL backlights consume less energy than other backlight technologies, which is especially important in battery powered portable electronics.

The structures of SSL sources used for general lighting and display backlights often are similar. In a common structure, these devices consist of a blue LED and at least one photoluminescent material to provide one or more additional wavelengths. When energized, some photons emitted by the LEDs are absorbed by the luminescent material and produce secondary photons of different wavelengths through the process of photoluminescence (PL). The light produced by the SSL source is a mixture of the emissions from the blue LED and the photoluminescent material. A variety of luminescent materials can be used in these applications including phosphors and luminescent nanomaterials.

Luminescent nanomaterials are comprised of semiconductor nanocrystals like spherical quantum dots and elongated quantum rods and inorganic nanophosphors. Semiconductor nanocrystals with sizes typically below 10 nm show size-tunable optical properties (size-dependent band gap and hence, size-dependent onset of absorption and spectral position of the emission band or emission colour) and electrochemical properties (size-dependent energetic positions of the valence and conduction band and hence, redox potentials of the charge carriers) due to particle size-dependent quantum confinement effects. Particularly favourable are their broad absorption bands (increasing absorption for all wavelengths shorter than the onset of absorption), their narrow emission bands (often revealing a symmetric shape), their high photoluminescence quantum yields and excellent photostability.

Light-emitting phosphors can also be used for lighting and display applications and in some instances phosphors with particle diameters less than 100 nm (i.e. nanoparticles) can be used. Such inorganic nanomaterials (also termed nanophosphors) include materials such as YAG:Ce. These nanophosphors are characterized by broad absorption bands, broad emission bands, good photoluminescence quantum yields and a high photostability. The spectral position of the absorption and emission of inorganic nanophosphors is not affected by size, but the scattering properties will have a size dependence. However, the enhanced surface-to-volume ratio with decreasing particle size can favour luminescence quenching at surface defects, thereby affecting the photoluminescence quantum efficiency and PL decay behaviour and rendering both properties size-dependent.

Other nanomaterials like dye doped or labelled polymer nanoparticles, inorganic particles or hybrid organic–inorganic nanoparticles are commonly not used for such applications and are beyond the scope of this document.

Generally, luminescent nanomaterials used in lighting and display applications are classified according to excitation spectrum, emission spectrum (including a specific emission wavelength peak and a narrow emission peak shape as measured by the full-width at half maximum (FWHM)), quantum efficiency, chemistry and others. Generally, these properties are achieved in a monodisperse material, with particles of similar sizes (allowing for manufacturing tolerances). Imparting multiple colours to a lighting or display product may involve the use of nanomaterials of multiple sizes, each of which may be specified individually. As a result of the properties of luminescent nanomaterials, lighting and display devices incorporating these materials can have excellent luminous efficacy and extraordinary colour quality.

This document codifies the format for specifying, reporting, and validating the essential properties of luminescent nanomaterials for use in lighting and display products.

NANOMANUFACTURING – MATERIAL SPECIFICATIONS –

Part 4-1: Luminescent nanomaterials – Blank detail specification

1 Scope

This part of IEC 62565, which is a Technical Specification, establishes a blank detail specification and format for listing essential optical and certain other characteristics of monodisperse luminescent nanomaterials. This document does not address mixtures or agglomerations of luminescent nanomaterials.

In addition, this document enables the customer to specify requirements in a standardized manner and to verify through standardized methods that the luminescent nanomaterial meets the required properties.

Numeric values to be specified for the properties and characteristics in this document are intentionally left blank and are determined by agreement between customer and material supplier. Properties and characteristics deemed by the customer or supplier as not relevant to a specific application are classified as “not applicable” or “not specified”.

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.

IEC 62607-3-1, *Nanomanufacturing – Key control characteristics – Part 3-1: Luminescent nanomaterials – Quantum efficiency*

IEC TS 62607-3-2, *Nanomanufacturing – Key control characteristics – Part 3-2: Luminescent nanoparticles – Determination of mass of quantum dot dispersion*

3 Terms, definitions and abbreviated terms

3.1 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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

- IEC Electropedia: available at http://www.electropedia.org/
- ISO Online browsing platform: available at http://www.iso.org/obp

3.1.1
absorption coefficient
corresponding absorbance divided by the optical path length through the sample

Note 1 to entry: For the purposes of this document, absorption coefficient is determined at a known concentration and at a wavelength where the impact of optical scattering is negligible.

3.1.2
colour
optical characteristic of a luminescent nanomaterial uniquely characterized by means of three coordinates in a colour space

Note 1 to entry: Examples of coordinates are the 1931 CIE tristimulus values and the CIELAB 1976 L*a*b* colour space.

Note 2 to entry: For determination of colour, it is also necessary to specify the illuminant (e.g. Illuminant A, Illuminant D65) and observer (e.g. 2° or 10°).

3.1.3
date of manufacture
date on which the luminescent nanomaterials were originally synthesized

3.1.4
emission spectrum
spectral distribution of the radiation emitted by a luminescent material for a specified excitation

[SOURCE: CIE S 017/E:2011, *ILV: International Lighting Vocabulary*, definition 17-380]

3.1.5
emission wavelength peak
wavelength at which the maximum emission occurs

3.1.6
emission wavelength range
range of wavelengths at which emission occurs

Note 1 to entry: To avoid contributions from stray light, the emission wavelength range is given at the wavelengths where the emission exceeds 5 % of the emission wavelength peak intensity.

3.1.7
excitation wavelength
specific wavelength used to stimulate a luminescent nanomaterial to emit light

3.1.8
FWHM
full-width at half maximum
range of emission wavelengths over which the emission spectrum intensity is greater than 50 % of its maximum value

3.1.9
ID number
manufacturing process identifier (3.1.11) that uniquely identifies the specific synthesis procedure or recipe used to synthesize the luminescent nanomaterials

3.1.10
luminescent nanomaterial
nanomaterial which emits light when excited by electrical, optical or other type of excitation

EXAMPLE quantum dots, nanophosphors

3.1.11
manufacturing process identifier
unique means for identifying a manufacturing process, indicating a specific set of process parameters

3.1.12
nanomaterial
material with any external dimension in the nanoscale or having an internal structure or surface structure in the nanoscale

[SOURCE: ISO/TS 80004-1:2015, 2.4]

3.1.13
peak absorbance
wavelength at which maximum electromagnetic radiation absorption occurs

3.1.14
polarization anisotropy
emission anisotropy
r
polarization sensitivity of a fluorescent sample

Note 1 to entry: Polarization anisotropy is defined in terms of the measured fluorescence intensity in the directions parallel, $I_{\parallel}$, and perpendicular, $I_{\perp}$, to the plane of incidence, but compared to the total fluorescent intensity, I_{T}, where:

$$r = \frac{I_{\parallel} - I_{\perp}}{I_{\mathrm{T}}} = \frac{I_{\parallel} - I_{\perp}}{I_{\parallel} + 2I_{\perp}}$$

3.1.15
quantum efficiency
efficiency of photon emission from luminescent nanomaterials after excitation

[SOURCE: IEC 62607-3-1:2014, 3.13, modified – In the definition, "nanoparticles" has been replaced by "nanomaterials after excitation".]

3.2 Abbreviated terms

C_{pk}	manufacturing process capability index
CVD	chemical vapour deposition
DLS	dynamic light scattering
EDX	energy dispersive X-ray spectroscopy
ICP	inductively coupled plasma
IR	infrared spectroscopy
MS	mass spectrometry
NIR	near-infrared spectroscopy
OES	optical emission spectroscopy
PFS	polarized fluorescence spectroscopy
PL	photoluminescence
PVD	physical vapour deposition
SAXS	small angle X-ray scattering
SEM	scanning electron microscopy
TGA	thermogravimetric analysis
TOPO	trioctylphosphine oxide
TEM	transmission electron microscopy
UV-Vis	ultraviolet-visible spectroscopy
XRD	X-ray diffraction

XRF X-ray fluorescence

YAG:Ce cerium-doped yttrium aluminium garnet

4 General introduction regarding measurement methods

The specification of material parameters of luminescent nanomaterials shall refer to measurement methods for which currently either no standards exist yet or for which such standards have recently been developed in a joint working group of IEC TC 113 and ISO/TC 229. For reasons of practicality for industrial use in manufacturing of nano-enabled electrotechnical products, this document recommends appropriate measurement methods for each material parameter.

In the absence of adequate standardized methods for industrial use, the user shall fulfil the following minimum documentation requirements:

- description of the sample preparation;
- measurement procedure;
- sample size and statistical significance;
- description of how the original measurement data are converted to the specified material parameter(s).

The choice of measurement methods and procedures shall be made with respect to the application of the material, taking into account their cost, robustness and efficiency. It is recommended that such methods are agreed upon between vendor and user, taking into consideration whether standards that address these methods are available.

5 Basic specification requirements

Luminescent nanomaterials produced in compliance with this document shall be qualified through routine process checks (in the manufacturing process of the material), demonstrating that the process is in a state of control. In accordance with common industry practice, the demonstration of control is implemented via statistical process control (SPC), as stipulated, for example, in IATF 16949:2016.

NOTE State of control implies that the process is under statistical process control with a defined manufacturing process capability index (C_{pk}).

6 Recommended specification format

6.1 General procurement

General information about a luminescent nanomaterial should be provided by the manufacturer or product specifier according to Table 1.

Table 1 – Format for general information

Item No.	Item		Information
1.1	Supplier		
1.2	Trade name		
1.3	Date of manufacture		
1.4	ID number		
1.5	Batch number		
1.6	Serial number		
1.7	Growth method		❑ Colloidal synthesis ❑ PVD ❑ CVD ❑ Others, specify:
1.8	Functionalization		❑ General classes ❑ TOPO ❑ Amines ❑ Carboxylic acids ❑ Phosphonic acid ❑ Other (specify):
1.9	Dispersion agent		❑ Solution (specify solvent): ❑ Solid (specify matrix):
1.10	Dispersion method		
1.11	Specification	Number	
		Revision level	
		Date of issue	
NOTE General procurement specification number, revision level, and part number/revision are specified by either the customer or luminescent nanomaterial supplier.			

6.2 Luminescent nanomaterial key control characterization

6.2.1 Physical and chemical key control characteristics

The specification format for physical and chemical key control characteristics of all luminescent nanomaterials shall be reported according to Table 2.

Table 2 – Physical and chemical key control characteristics

Item No.	Item	Specification	Specified measurement method	Other measurement methods
2.1	Size	Nominal [ ] ± Tolerance [ ] nm	TEM	UV-Vis; XRD; DLS, Fluid Flow Fractionation, SAXS
2.2	Shape		TEM	
2.3	Polarization anisotropy		PFS	
2.4	Mass		IEC 62607-3-2	
2.5	Basic composition			Descriptions for core, shell and ligand under development
2.6	Inorganic content mass fraction	Mass fraction is no greater than: [ ]	TGA	NIR; ICP-MS; ICP-OES; XPM;
2.7	Metal content mass fraction	Mass fraction is not greater than: [ ]	ICP-MS	ICP-OES; XRF, Electrochemistry
2.8	Presence of cadmium	Mass fraction is no greater than: [ ]	ICP-MS	ICP-OES, XRF, Electrochemistry
2.9	Presence of lead	Mass fraction is no greater than: [ ]	ICP-MS	ICP-OES, XRF, Electrochemistry
2.10	Other impurities	Mass fraction is no greater than: [ ]	ICP-MS	ICP-OES, XRF

6.2.2 Optical key control characteristics

The specification format for optical key control characteristics of all luminescent nanomaterials shall be according to the colour of light emission as given in Table 3. The excitation wavelength shall be given for these key control characteristics.

NOTE Luminescent nanomaterials can also be excited electrically; however, electrical key control characteristics are not specified in this document.

Table 3 – Optical key control characteristics by emission colour

Property	Blue	Cyan	Green	Yellow	Orange	Red	Deep red	Specified measurement method	Other measurement method
Colour	—	—	—	—	—	—	—	Absorbance spectroscopy	Fluorescence spectroscopy
Absorption coefficient (cm^{-1})								Absorbance spectroscopy	
Excitation wavelength (nm)								Fluorescence spectroscopy	
Emission wavelength peak (nm)								Fluorescence spectroscopy	
Emission wavelength range (nm)								Fluorescence spectroscopy	
FWHM (nm)								Fluorescence spectroscopy	
Quantum efficiency								IEC 62607-3-1	

NOTE 1 Specified methods are under investigation within a joint working group of ISO/TC 229 and IEC TC 113.

NOTE 2 Nominal values and tolerances to be provided (see 3.1.2).

7 An overview of test methods and analysis techniques

NOTE Test methods are the subject of a joint working group of ISO/TC 229 and IEC TC 113. The information given here is for overview only and will adapt to results from the joint working group as they become available.

Accurately measuring and characterizing the quality of luminescent nanomaterials are crucial for the continued growth of nanomaterials in general lighting and display applications. Significant differences in both methodology and interpretation continue to exist from one measurement laboratory to another. For this reason, comparison and specification of the quality of luminescent nanoparticle materials is extremely difficult. While progress in these measurements is being been made, significant improvements are still needed to accurately measure and characterize material quality.

The most extensively utilized techniques for luminescent nanomaterials include UV-Vis spectroscopy, fluorescence spectroscopy, ICP-MS, TGA, SEM, and TEM. Fluorescence spectroscopy is also termed fluorimetry and is a particular type of emission spectroscopy. Table 4 summarizes the techniques commonly used for analysing some specific key control properties.

Thermogravimetric analysis quantitatively determines the amounts of inorganic and organic materials in bulk samples.

TEM images give a rough idea of quality, while higher-resolution TEM images can monitor the shape of individual particles.

For a qualitative analysis of the morphology of a luminescent nanomaterial sample, TEM should be used. The methodology by which TEM images are selected should always be specified. For quantitative estimation of sample purity, a combination of ICP-MS, TGA, IR, Raman, and NIR methods is recommended. For determination of optical properties, UV-Vis should be used to determine properties related to light absorption and fluorescence spectroscopy should be used to determine properties related to excitation and emission.

Table 4 – Summary of test methods

Property	Method						
	SEM/EDX	TEM	UV-Vis	Fluorescence spectroscopy	TGA	ICP-MS	IR, NIR, Raman
Morphology	Secondary	Primary					
Purity					Secondary	Primary	Secondary
Length and diameter	Secondary	Primary	Secondary	Secondary			
Peak absorbance			Primary	Secondary			
Absorbance spectrum			Primary				
Excitation wavelength			Secondary	Primary			
Emission spectrum				Primary			