IEC TS 62607-3-2

Edition 1.0 2017-01

TECHNICAL SPECIFICATION

colour inside

Nanomanufacturing – Key control characteristics – Part 3-2: Luminescent nanoparticles – Determination of mass of quantum dot dispersion

IEC TS 62607-3-2:2017-01(en)

IEC Central Office
3, rue de Varembé
CH-1211 Geneva 20
Switzerland

Tel.: +41 22 919 02 11
Fax: +41 22 919 03 00
info@iec.ch
www.iec.ch

About the IEC
The International Electrotechnical Commission (IEC) is the leading global organization that prepares and publishes International Standards for all electrical, electronic and related technologies.

About IEC publications
The technical content of IEC publications is kept under constant review by the IEC. Please make sure that you have the latest edition, a corrigenda or an amendment might have been published.

IEC Catalogue - webstore.iec.ch/catalogue
The stand-alone application for consulting the entire bibliographical information on IEC International Standards, Technical Specifications, Technical Reports and other documents. Available for PC, Mac OS, Android Tablets and iPad.

IEC publications search - www.iec.ch/searchpub
The advanced search enables to find IEC publications by a variety of criteria (reference number, text, technical committee,…). It also gives information on projects, replaced and withdrawn publications.

IEC Just Published - webstore.iec.ch/justpublished
Stay up to date on all new IEC publications. Just Published details all new publications released. Available online and also once a month by email.

Electropedia - www.electropedia.org
The world's leading online dictionary of electronic and electrical terms containing 20 000 terms and definitions in English and French, with equivalent terms in 16 additional languages. Also known as the International Electrotechnical Vocabulary (IEV) online.

IEC Glossary - std.iec.ch/glossary
65 000 electrotechnical terminology entries in English and French extracted from the Terms and Definitions clause of IEC publications issued since 2002. Some entries have been collected from earlier publications of IEC TC 37, 77, 86 and CISPR.

IEC Customer Service Centre - webstore.iec.ch/csc
If you wish to give us your feedback on this publication or need further assistance, please contact the Customer Service Centre: csc@iec.ch.

IEC TS 62607-3-2

Edition 1.0 2017-01

TECHNICAL SPECIFICATION

colour inside

Nanomanufacturing – Key control characteristics – Part 3-2: Luminescent nanoparticles – Determination of mass of quantum dot dispersion

INTERNATIONAL ELECTROTECHNICAL COMMISSION

ICS 07.120

ISBN 978-2-8322-3767-0

Warning! Make sure that you obtained this publication from an authorized distributor.

CONTENTS

FOREWORD 3
INTRODUCTION 5
1 Scope 7
2 Normative references 7
3 Terms and definitions 7
4 Apparatus 7
5 Preparation of sample 8
6 Procedure 8
6.1 General 8
6.2 Heating 8
6.3 Mass concentration of additional samples 8
6.4 Potential instrument errors 9
6.4.1 Static 9
6.4.2 Bumping 9
7 Results and calculations 9
7.1 Concentration and mass of QDs 9
7.2 Mass concentration from relative absorbance 9
8 Measurement uncertainty 9
9 Test report 10
Annex A (informative) Example of determination of mass of QD dispersion 11
Annex B (informative) Determination of mass concentration of additional samples 12
B.1 General 12
B.2 Preparation 12
B.3 Results and calculations 12
Bibliography 13

Figure A.1 – Mass of QD dispersion vs. temperature 11

INTERNATIONAL ELECTROTECHNICAL COMMISSION

____

NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 3-2: Luminescent nanoparticles – Determination of mass of quantum dot dispersion

FOREWORD

1) The International Electrotechnical Commission (IEC) is a worldwide organization for standardization comprising all national electrotechnical committees (IEC National Committees). The object of IEC is to promote international co-operation on all questions concerning standardization in the electrical and electronic fields. To this end and in addition to other activities, IEC publishes International Standards, Technical Specifications, Technical Reports, Publicly Available Specifications (PAS) and Guides (hereafter referred to as "IEC Publication(s)"). Their preparation is entrusted to technical committees; any IEC National Committee interested in the subject dealt with may participate in this preparatory work. International, governmental and non-governmental organizations liaising with the IEC also participate in this preparation. IEC collaborates closely with the International Organization for Standardization (ISO) in accordance with conditions determined by agreement between the two organizations.
2) The formal decisions or agreements of IEC on technical matters express, as nearly as possible, an international consensus of opinion on the relevant subjects since each technical committee has representation from all interested IEC National Committees.
3) IEC Publications have the form of recommendations for international use and are accepted by IEC National Committees in that sense. While all reasonable efforts are made to ensure that the technical content of IEC Publications is accurate, IEC cannot be held responsible for the way in which they are used or for any misinterpretation by any end user.
4) In order to promote international uniformity, IEC National Committees undertake to apply IEC Publications transparently to the maximum extent possible in their national and regional publications. Any divergence between any IEC Publication and the corresponding national or regional publication shall be clearly indicated in the latter.
5) IEC itself does not provide any attestation of conformity. Independent certification bodies provide conformity assessment services and, in some areas, access to IEC marks of conformity. IEC is not responsible for any services carried out by independent certification bodies.
6) All users should ensure that they have the latest edition of this publication.
7) No liability shall attach to IEC or its directors, employees, servants or agents including individual experts and members of its technical committees and IEC National Committees for any personal injury, property damage or other damage of any nature whatsoever, whether direct or indirect, or for costs (including legal fees) and expenses arising out of the publication, use of, or reliance upon, this IEC Publication or any other IEC Publications.
8) Attention is drawn to the Normative references cited in this publication. Use of the referenced publications is indispensable for the correct application of this publication.
9) Attention is drawn to the possibility that some of the elements of this IEC Publication may be the subject of patent rights. IEC shall not be held responsible for identifying any or all such patent rights.

The main task of IEC technical committees is to prepare International Standards. In exceptional circumstances, a technical committee may propose the publication of a Technical Specification when

- the required support cannot be obtained for the publication of an International Standard, despite repeated efforts, or
- 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 62607-3-2, which is a Technical Specification, has been prepared by IEC technical committee 113: Nanotechnology standardization for electrical and electronic products and systems.

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

Enquiry draft	Report on voting
113/243/DTS	113/348/RVC

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 of the IEC 62607 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 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

- transformed into an International Standard,
- reconfirmed,
- withdrawn,
- replaced by a revised edition, or
- amended.

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

IMPORTANT – The 'colour inside' logo on the cover page of this publication indicates that it contains colours which are considered to be useful for the correct understanding of its contents. Users should therefore print this document using a colour printer.

INTRODUCTION

Quantum dots (QDs) are crystals of semiconductor materials measuring typically but not limited to 2 nm to 15 nm in a given dimension and could exhibit desirable absorption and emission properties. QDs can be synthesized by a variety of methods, the colloidal method being the most common. Through the colloidal method, QDs are synthesized in the presence of stabilizing ligands to maintain colloidal stability. These ligands can remain present after purification and are an important component of QD dispersibility and stability and can impact the optical properties. Often, QD samples are provided as a stable dispersion instead of a dried powder. The method specified in this document provides a means to determine the mass of QDs in that dispersion.

In addition to the semiconductor material and the tightly bound stabilizing ligands, QD dispersions could potentially contain unreacted precursors, side-products of the precursors, and free ligands. Due to the difficulty of removing and determining the amount of these impurities, the dried mass is not sufficient for measuring QD mass. Instead, a method is chosen that will remove or decompose the impurities while leaving the semiconductor material intact.

One method to volatilize or decompose the impurities and surfactant ligands is by heating the QD sample to high temperatures (650 °C). Heating QD samples to such high temperatures is typically destructive to the QD sample, so this analysis would need to be performed on a small portion that is representative of the entire sample.

Thermogravimetric analysis (TGA) is a convenient tool for accurately measuring mass loss of a small amount of sample (10 mg to 50 mg) when heated to high temperatures under either an N_2 or O_2 environment. The general procedure is to add an amount of dispersed material to the TGA pan, remove the dispersing solvent, and heat the pan to 650 °C in the TGA. The leftover mass is mostly the inorganic content of the QD material. Compared to other instrumentation such as inductively coupled plasma mass spectrometry, TGA is convenient, ubiquitous, and can give a direct measure of the inorganic mass of a quantum dot mixture.

The following are potential errors to the TGA-based method:

- Under an N_2 environment, it is possible that instead of volatilizing, the impurities will decompose and be converted into graphite or another non-volatile product. This will result in a measured mass that is greater than just the mass of the semiconductor material, whereas heating organic precursors to 650 °C typically results in only a minimal amount of residual mass (< 2 % of initial mass).
- Under an O_2 environment, heating samples can result in added mass from oxidation of metal and chalcogenide species. To avoid this complication, samples are heated under an N_2 environment.
- Inorganic-based impurities can leave a residual mass upon heating. These impurities (such as carboxylate salts, phosphonate salts) are typically not soluble in commonly used dispersing solvents (hexanes, toluene) and the concentration of these impurities in the dispersion is expected to be low, if a proper solvent is used, unless they are being solubilized by the QD material. If a solvent for potential impurities is used for the dispersion, then additional analysis may be necessary to determine the level of inorganic impurities. For safety purposes, this method should not be used for solutions containing perchlorate salts as this may cause an explosion.
- Tightly bound surface ligands may not fully decompose under these conditions. It is possible that the surface ligands would be converted into graphite instead of volatilizing. In this case, the residual ligand mass would be included in the mass calculation.
- Certain metal or main group elements may volatilize from the quantum dot depending on the chemical composition and heating conditions. Under those circumstances, a change in the maximum temperature is made. These methods have worked well for QD dispersions containing CdSe, CdS, ZnS, InP, InAs, PbS and mixtures thereof.

This document offers a reliable method of determining the mass of a colloidal QD dispersion after removing or decomposing impurities while minimizing potential errors that may otherwise be encountered.

NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 3-2: Luminescent nanoparticles – Determination of mass of quantum dot dispersion

1 Scope

This part of IEC 62607 specifies a method for determining the mass of a sample of QD dispersion after the removal of impurities and surfactant ligands through heating at high temperatures.

2 Normative references

There are no normative references in this document.

NOTE The Bibliography lists documents that are useful for its application.

3 Terms and definitions

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

3.1
portion
<QD dispersion> small amount of the sample to be analysed that is representative of the concentration of the sample

3.2
dispersing solvent
solvent used to disperse the quantum dots

Note 1 to entry: Ideally, the dispersing solvent has a boiling point between 60 °C and 120 °C.

Note 2 to entry: Common solvents include toluene and hexanes.

4 Apparatus

The apparatus shall consist of the following:

- Hot plate, heat gun, or other heating apparatus for removing dispersing solvent.
- Vacuum line and a chamber to contain the TGA pan for removing dispersing solvent.
- Gas flow for removing dispersing solvent.
- Moderate pressure line (typically < 3,45 kPa) for flow of an inert gas (for example, N_2, Ar, He) over the surface of the dispersing solvent. A sufficient flow shall be maintained to volatilize the solvent, but not so high as to potentially result in spilling the sample from the pan, thus invalidating the measurement.
- Thermogravimetric analysis instrument for performing reliable thermogravimetric analysis.
- TGA pan, typically a small vessel that is compatible with the temperature ranges and design of the thermogravimetric analysis instrument used.
- Syringe or calibrated pipette.