

TECHNICAL SPECIFICATION



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

INTERNATIONAL
ELECTROTECHNICAL
COMMISSION

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

**NANOMANUFACTURING –
KEY CONTROL CHARACTERISTICS –****Part 3-2: Luminescent nanoparticles –
Determination of mass of quantum dot dispersion**

FOREWORD

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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- withdrawn,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.

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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 N₂ or O₂ 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 N₂ 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 O₂ environment, heating samples can result in added mass from oxidation of metal and chalcogenide species. To avoid this complication, samples are heated under an N₂ 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.