IEC TS 62607-4-7

Edition 1.0 2018-08

TECHNICAL SPECIFICATION

colour inside

Nanomanufacturing – Key control characteristics –
Part 4-7: Nano-enabled electrical energy storage – Determination of magnetic impurities in anode nanomaterials, ICP-OES method

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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 62607-4-7, 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
113/405/DTS	113/430/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 TS 62607 series, published under the general title *Nanonmanufacturing – 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- withdrawn,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

Magnetic impurities often have significant influence on the performance of anode nanomaterials for nano-enabled electrical energy storage. If anode nanomaterials have high magnetic impurity content, it may lead to serious self-discharge and degrade the performance and cycle life. The magnetic impurities are also easy to aggregate, which can cause safety issues such as internal short-circuit fire, or even explosion. Therefore, accurate evaluation of magnetic impurity content is important for the safety and performance of energy storage devices. [1,2,3][1]

Inductively Coupled Plasma Optical Emission Spectrometer (ICP-OES) method is a mature and precise method to measure the concentration of metal elements in the tested materials, but the method to gather the magnetic substance has not been standardized. This document introduces a reliable method for gathering and determining the magnetic impurity content in the anode nanomaterials of an energy storage device. It will help to better control the quality of electrode nanomaterials.

This document introduces a determination of magnetic impurities using ICP-OES method for the electrochemical characterization of nano-enabled anode materials for electrical energy storage devices.

This standardized method is intended for use in comparing the characteristics of anode nanomaterials in the study stage, not for evaluating the electrode in end products.

The method is applicable to materials exhibiting function or performance only possible with nanotechnology, intentionally added to the active materials to measurably and significantly change the reliability or one or more physical properties of electrical energy storage devices.

In this context it is important to note that the percentage content of nanomaterials of the device in question has no direct relation to the applicability of this document, because minute quantities of nanomaterials are frequently sufficient to improve the performance significantly.

The fraction of nanomaterials in electrodes or electrode coatings is not of relevance for using this method.

1 Numbers in square brackets refer to the Bibliography.

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WARNING – Persons using this method should be familiar with normal laboratory practice. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

IMPORTANT – It is absolutely essential that tests conducted according to this document be carried out by suitably trained staff.

1 Scope

This part of IEC TS 62607 provides a method for the determination of magnetic impurities in anode nanomaterials for energy storage devices using an Inductively Coupled Plasma Optical Emission Spectrometer (ICP-OES), including test overview, reagents, apparatus, test procedures, test results and test report.

This document applies to the determination of the total content of magnetic impurities (iron, cobalt, chromium, and nickel) ≥ 0,02 mg/kg which can be attracted by magnet.

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 3696:1987, *Water for analytical laboratory use – Specification and test methods*

ISO 6353-1:1982, *Reagents for chemical analysis – Part 1: General test methods*

ISO 18842:2015, *Aluminium oxide primarily used for the production of aluminium – Method for the determination of tapped and untapped density*

3 Terms and definitions

No terms and definitions are listed in this document.

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

4 Overview of the measurement

First, the magnetic impurities of samples are attracted by a magnet. And then the magnetic impurities are dissolved in acidic solution. Lastly ICP-OES is used to determine the content of iron, cobalt, chromium, and nickel.

5 Reagents

5.1 Nitric acid

$c(HNO_3)$ = 15 mol/L, $\rho \approx$ 1,40 g/mL. It shall be analytical reagent grade.

5.2 Hydrochloric acid

$c(HCl)$ = 12 mol/L, $\rho \approx$ 1,18 g/mL. It shall be analytical reagent grade.

5.3 Nitrohydrochloric acid

Solution obtained by mixing one volume of nitric acid with three volumes of hydrochloric acid.

5.4 Ethanol

$w(C_2H_6O) \geq$ 99,7 %. It shall be analytical reagent grade.

5.5 Argon gas

It shall have volume fraction of not less than 99,99 %.

5.6 Standard solutions

5.6.1 Iron standard solution

In accordance with ISO 6353-1:1982, concentration 0,001 g/mL.

5.6.2 Cobalt standard solution,

In accordance with ISO 6353-1:1982, concentration 0,001 g/mL.

5.6.3 Chromium standard solution,

In accordance with ISO 6353-1:1982, concentration 0,001 g/mL.

5.6.4 Nickel standard solution,

In accordance with ISO 6353-1:1982, concentration 0,001 g/mL.

Subject to specific instructions, pure reagents and grade 3 water as specified in ISO 3696:1987 shall be used in the process.

6 Apparatus

6.1 ICP-OES

The technique ICP-OES is based on the measurement of emission at one wavelength highly selective for a specific element. For the determination of magnetic impurities, therefore, the test wavelengths are chosen in accordance with Table 1.

Table 1 –Recommended test wavelengths for the determination of magnetic impurities

Metal element	Wavelength nm
iron	238,204
cobalt	228,616
chromium	267,716
nickel	231,604

6.2 Conical flask

The conical flask is transparent and has a volume of 250 mL.

6.3 Sample tank

The sample tank is made of plastic material and has a volume of 500 mL, with inner and outer cover. The sample tank should be good sealing. Its calibre is consistent with the calibre of conical flasks.

6.4 Magnet

The magnet has 0,6 T of magnetic field intensity (allowable deviation within 5 %). Its diameter is 15 mm to 20 mm and its length is 45 mm to 55 mm. The magnet should be completely encapsulated with polytetrafluoroethylene materials, resistant to strong acid and alkali.

NOTE The magnetic field intensity (0,6 T) used in this document refers to the maximum magnetic field intensity of the magnet, which is probed at the surface of the magnet by the magnetometer.

6.5 Electronic balance

The balance has a resolution of 0,001 g.

6.6 Scroll equipment

The scroll mode of the equipment is shown in Annex A. The range of rotational speed is 60 rev/min to 100 rev/min.

6.7 Ultrasonic cleaning equipment

An ultrasonic cleaning device commonly used in the laboratory is applied to this document.

6.8 Heating device

The heating device has a capability for adjustment of heating temperature to boil aqueous solution, such as a hot plate or electric furnace.

7 Preparation of series of standard solutions

Prepare a series of standard solutions, and make sure that the concentration of the sample solution is in the linear range of the calibration curve. Beside blank solution, not less than three calibration standard solutions are prepared.