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TECHNICAL SPECIFICATION

colour inside

Nanomanufacturing – Key control characteristics – Part 4-5: Cathode nanomaterials for nano-enabled electrical energy storage – Electrochemical characterization, 3-electrode cell 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 62607-4-5, 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
113/317/DTS	113/342/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 in 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- withdrawn,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

The future utilization of renewable energy technologies depends significantly on the development of efficient systems for energy storage. Conventional approaches exist for the storage of electrical energy from stationary power plants, currently fuelled by many new ideas in conjunction with the emerging "Smart Grid". For future e-mobility for individual transportation there is only one attractive solution: a battery that can store enough energy to allow all-electric driving with a range of several hundred kilometres. The current solutions already on the market can only be seen as temporary solutions. From today's perspective, lithium-ion batteries and their derivative innovative concepts are regarded as the most promising candidates. Electrodes made from nanoscale composites will play a key role in the future. Innovative materials will be developed and systematically optimized, which implies testing of a large number of different materials.

Characterization of the electrochemical properties of cathode nanomaterials used in electrical energy storage devices is important for their customized development. This document provides a standard methodology which can be used to characterize the electrochemical properties of new cathode nanomaterials that will be employed in electrical energy storage devices. Following this method will allow comparison of different types of cathode nanomaterial and comparing the results of different research groups.

This document introduces a 3-electrode cell method for the electrochemical characterization of nano-enabled cathode materials for electrical energy storage devices.

This standardized method is intended for use in comparing the characteristics of cathode nanomaterials in the development 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 capacity of electrical energy storage devices.

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

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

NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 4-5 Cathode nanomaterials for nano-enabled electrical energy storage – Electrochemical characterization, 3-electrode cell method

1 Scope

This part of IEC 62607 provides a standardized method for the determination of electrochemical properties of cathode nanomaterials such as lithium iron phosphate (LFP) for electrical energy storage devices. This method will enable the industry to:

a) decide whether or not a cathode nanomaterial is usable, and
b) select a cathode nanomaterial suitable for their application.

This document includes:

- recommendations for sample preparation,
- outlines of the experimental procedures used to measure cathode nanomaterial properties,
- methods of interpretation of results and discussion of data analysis, and
- case studies.

NOTE The very purpose of this method is to arrive at a detailed characterization of the electrodes so that individual contribution of the anode and cathode for performance and degradation could be predicted. The method can be applied for characterization of the electrode working as cathode or/and as anode.

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/TS 80004-1, *Nanotechnologies – Vocabulary – Part 1: Core terms*

3 Terms, definitions and abbreviated terms

3.1 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO/TS 80004-1 and the following 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
cathode nanomaterial
material used as a cathode in a nano-enabled energy storage device which contains a fraction of nanomaterial and exhibits function or performance made possible only with the application of nanotechnology

Note 1 to entry: The cathode is a multilayered foil consisting of (1) an aluminium current collector, (2) an optional adhesion promoting carbon layer (to enhance cathode layer adhesion if necessary) and (3) the cathode layer. This cathode layer consists of the active phase (e.g. lithium containing mixed oxides or phosphate, such as LFP), a conducting phase (carbon black) and an organic binder (PVDF).

3.1.2
anode material
material used as counter electrode (CE) for the electrochemical characterization of cathodes in the 3-electrode cell

Note 1 to entry: The anode may be a tape-cast graphite electrode consisting of (1) a copper current collector foil and (2) the active layer composed of graphite, a conducting phase (carbon black) and organic binder (PVDF, CMC). Alternatively, metallic lithium may be utilized as CE. Using lithium, the necessity of balancing the capacities of cathode and anode is dropped. However, the cycling stability of lithium is strongly limited in comparison to a graphite anode. Thus the choice of the counter electrode has to be adapted to the purpose of the investigation.

3.1.3
reference electrode
RE
electrode not actively involved in the battery cell reactions (charging, discharging)

Note 1 to entry: The reference electrode is placed in the cell arrangement to enable the measurement of the electrode potentials of cathode and anode. Both values are determined with respect to the RE's potential. To ensure a proper measurement, the reference electrode's potential has to be held constant. Thus a currentless contacting of this electrode is realized to prevent the formation of overpotentials. In lithium ion battery research the most common reference electrode material is metallic lithium in contact with a lithium ion containing electrolyte.

3.1.4
3-electrode screw cell
cell providing the geometrical conditions in the three electrode arrangement

Note 1 to entry: The independent electrochemical investigation of cathode and anode material is carried out in 3-electrode screw cells. This notion describes a cell in three electrode arrangement providing battery-like geometrical conditions of cathode and anode. Additionally, a lithium reference electrode is included to enable the determination of the individual electrode potentials rather than of the overall cell voltage. The cell setup includes springs and metallic current collectors and the electrode package with anode/separators + electrolyte/cathode. The reference electrode is placed between anode and cathode, separated from these electrodes by layers of separator. For this purpose, various cell designs are possible. The case study in Annex A shows a cell design based on a half-inch PFA Swagelok fitting.[1]

3.1.5
cell voltage
U_{cell}
difference of the electrochemical potentials of the cathode and the anode

3.1.6
electrode potential
difference between the electrochemical potential of an electrode in the 3-electrode cell and the potential of the RE

Note 1 to entry: Electrode potentials φ_{WE} and φ_{CE} are the differences between the electrochemical potential of the respective electrode in the 3-electrode cell and the potential of the RE. In case of intercalation electrode materials, it is determined by the lithiation state of the material. Additionally, the overpotential of the respective electrode reaction influences its potential. Consequently it is a valuable physical parameter to be determined, as it includes a significant amount of information on the individual electrodes and their actual state.

3.1.7
cell resistance
R_{el}
ohmic internal resistance of the testing cell

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1 PFA Swagelok fitting is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by IEC of this product.

Note 1 to entry: R_{el} is the sum of the ohmic resistivities (e.g. electrolyte, contact resistance) within the cell.

3.1.8
charge-discharge cycle
procedure which includes charging and discharging of the testing cell

Note 1 to entry: The freshly assembled cell is completely discharged. During charging, the lithium anode is biased negatively above the zero current potential, lithium cations are reduced and metallic lithium is deposited at the surface of the lithium anode. During galvanic discharge through an external circuit (load) metallic lithium is in turn oxidized at the anode, which shows a negative potential while the cathode potential is positive. Now metallic lithium oxidizes to lithium ions and dissolves in the electrolyte. Lithium ions incorporate into the crystal lattice of the cathode material. The charging/discharging processes are reversible within certain limits.

3.2 Abbreviated terms

LFP	lithium iron phosphate, $LiFePO_4$
C	graphite (layered modification of carbon)
Li	lithium
WE	working electrode, as cathode
CE	counter electrode, as anode
RE	reference electrode
PVDF	polyvinylidene fluoride
EC	ethylene carbonate
DEC	diethyl carbonate
$LiPF_6$	lithiumhexafluorophosphate (common conductive salt of lithium ion battery electrolytes)
PE	polyethylene
PFA	perfluoroalkoxy rubber
PEEK	polyether ether ketone rubber
OCV	open circuit voltage
OCP	open circuit potential
EIS	electrochemical impedance spectroscopy.

4 Sample preparation methods

4.1 General

For the electrochemical characterization of the cathode nanomaterial 3-electrode screw cells are used. The main aspects for preparation of these measuring cells are:

a) pre-treatment of the electrodes;
b) choice of the separator and selecting a proper electrolyte/ electrolyte volume;
c) applying a defined and valid pressure on the electrode/separator package;
d) mounting of reference electrode.

4.2 Reagents

4.2.1 Cathode foil

The cathode foil can be stored in a desiccator under low atmospheric moisture for the range of several days up to weeks. Previous to the assembly of the cell, cathode material is dried (see 4.3) and put into an argon filled glove box immediately after preparation to avoid contact with atmospheric moisture.