

TECHNICAL SPECIFICATION



**Nanomanufacturing – Key control characteristics –
Part 4-1 Cathode nanomaterials for lithium ion batteries – Electrochemical
characterisation, 2-electrode cell method**

IEC TS 62607-4-1:2014

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CONTENTS

FOREWORD.....	3
INTRODUCTION.....	5
1 Scope.....	6
2 Normative references	6
3 Terms, definitions, acronyms and abbreviations	6
3.1 Terms and definitions.....	6
3.2 Acronyms and abbreviations	7
4 Sample preparation methods	7
4.1 General.....	7
4.2 Reagents	8
4.2.1 Cathode foil	8
4.2.2 Anode.....	8
4.2.3 Solvents and separator	8
4.3 Pre-treatment of the cathode nanomaterial	8
4.4 Preparation of the screw cell.....	9
4.5 Disassembly of the screw cell	10
5 Measurement of electrochemical properties	10
5.1 General.....	10
5.2 Open circuit voltage (OCV)	10
5.2.1 Demarcation of method.....	10
5.2.2 Experimental procedures and measurement conditions.....	10
5.3 Potentiostatic electrochemical impedance spectroscopy (EIS)	10
5.3.1 Demarcation of method.....	10
5.3.2 Experimental procedures and measurement conditions.....	10
5.4 Charge discharge experiment (constant current constant voltage, CCCV)	10
5.4.1 Demarcation of method.....	10
5.4.2 Experimental procedures and measurement conditions.....	11
6 Data analysis / Interpretation of results.....	11
6.1 Open circuit potential	11
6.2 Electrochemical impedance spectroscopy	11
6.3 Constant current constant voltage (CCCV) charging-discharging	11
Annex A (informative) Case study	12
A.1 Sample preparation.....	12
A.2 Results for a LCO electrode	15
Figure A.1 – Components for the cell	12
Figure A.2 – Construction steps (a to g).....	14
Figure A.3 – Open circuit voltage/potential time graph	15
Figure A.4 – Electrochemical impedance graph.....	15
Figure A.5 – Constant current / constant voltage – Charge / Discharge.....	16
Figure A.6 – Capacity per cycle	17
Table 1 – Spring force and pressure	9

INTERNATIONAL ELECTROTECHNICAL COMMISSION

**NANOMANUFACTURING –
KEY CONTROL CHARACTERISTICS –****Part 4-1 Cathode nanomaterials for lithium ion batteries –
Electrochemical characterisation, 2-electrode cell method**

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 62607-4-1, 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
113/173/DTS	113/192/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 publication 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 publication will remain unchanged until the stability date indicated on the IEC web site under "<http://webstore.iec.ch>" in the data related to the specific publication. At this date, the publication 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 utilisation 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 regarded as temporary solutions. From today's perspective, lithium-ion batteries and their derivative innovative concepts must be 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 lithium ion batteries is important for their customized development. This IEC technical specification provides a standard methodology which can be used to characterize the electrochemical properties of new cathode nanomaterials that will be employed in lithium ion batteries. Following this method will allow comparison of different types of cathode nanomaterial and comparison of the results of different research groups.

A revised edition 2.0 is already under preparation to introduce changes proposed by IEC SC 21A. The future edition may e.g. include the following changes:

- The title will be amended: the term "lithium ion batteries" will be replaced by "nano-enabled electrical energy storage".
- The scope will be revised to change the phrase "lithium ion battery" to e.g., "lithium ion batteries utilizing lithium iron phosphate".
- The definition of "electrode nanomaterials" will be revised to be more specific, reading e.g. "electrode containing a nanomaterial portion of more than xx% by weight".

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NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 4-1 Cathode nanomaterials for lithium ion batteries – Electrochemical characterisation, 2-electrode cell method

1 Scope

This part of IEC 62607 provides a standardized method for the determination of electrochemical properties of lithium ion battery cathode nanomaterials to enable customers to:

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

This technical specification includes:

- definitions of terminology used in this document,
- 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,
- case studies and
- references.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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, acronyms and abbreviations

3.1 Terms and definitions

For the purposes of this document, the core terms and definitions of ISO/TS 80004-1 and the following terms and definitions apply.

3.1.1

cathode nanomaterial

electrodes used as cathodes in lithium ion batteries

Note 1 to entry: The cathode nanomaterial is a foil with a multilayered layout, built up 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 LCO, NCA, NCM, and LFP), a conducting phase (carbon black) and an organic binder (PVDF).

3.1.2

screw cell

cell providing the geometrical conditions in the two-electrode arrangement

Note 1 to entry: The electrochemical characterisation of the cathode nanomaterial is carried out in screw cells. The cell setup includes springs and metallic spacers and the electrode package with the anode, the separator impregnated with electrolyte and the cathode. For this purpose, various cell designs are possible. The case study in Annex A shows a cell design based on ½ inch PFA Swagelok fitting.¹

3.1.3 cell voltage

U_{cell}

difference of the electrochemical potentials of the cathode and the anode

3.1.4 cell resistance

R_{el}

ohmic internal resistance of the testing cell

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

3.1.5 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 oxidises 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 Acronyms and abbreviations

LCO: Lithium cobalt oxide LiCoO_2

NCA: Lithium nickel cobalt aluminium oxide $\text{Li}(\text{Ni}, \text{Co}, \text{Al})\text{O}_2$

NCM: Lithium nickel cobalt manganese oxide $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$

LFP: Lithium iron phosphate LiFePO_4

PVDF: Polyvinylidene fluoride

EC: ethylene carbonate

DEC: diethyl carbonate

PE: polyethylene

OCV: Open circuit voltage

4 Sample preparation methods

4.1 General

For the electrochemical characterisation of the cathode nanomaterial screw cells are used. The main aspects in preparation of these measuring cells are:

- pre-treatment of the electrodes,
- electing a proper electrolyte/ electrolyte volume, and
- applying a defined and valid pressure on the electrode package.

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.

4.2 Reagents

4.2.1 Cathode foil

The cathode material is put into an argon filled glove box immediately after preparation/ receiving to avoid contact to atmospheric moisture.

4.2.2 Anode

Metallic lithium is used as an anode material. The lithium foil (thickness $d = 0,25$ mm) should be unpacked in an argon filled glove box and then used as delivered.

4.2.3 Solvents and separator

The material testing should be carried out in an electrolyte of comparable composition. Currently LiPF_6 -containing electrolytes are usually applied in commercial batteries. For the investigation commercial electrolyte of the type LP40 (1M LiPF_6 in 1:1 EC:DEC) with a defined purity and water content < 5 ppm or equivalent is recommended. Use of the alternative electrolyte is possible, however in this case the wettability of separator and electrode material by alternative electrolyte should be proven in separate tests. Viledon^{®2}, a PE-nonwoven by company Freudenberg, is the chosen separator material. Other separator material can also be used, however in this case the wettability of separator electrolyte should be proven in separate tests.

4.3 Pre-treatment of the cathode nanomaterial

The cathode foil is dried in a vacuum oven to achieve water contents of < 100 ppm in the active material. Exemplary drying conditions are: $T = 120$ °C, $p = 1$ mbar – 5 mbar, $t = 12$ h.

It is suggested to control the water content of the cathode by drying to the constant mass. The drying procedure should be proven to achieve water content of < 100 ppm by Karl-Fischer titration for first five cathode samples. After that the drying to the constant mass can be applied as a standard.

The electrodes used in the Swagelok cell are punched out or laser cutted from the foil coated with cathode layer. The mass of the punched electrodes is determined by subtracting the mass of uncoated foil from the mass of coated foil.

From the mass of the electrodes the theoretical capacity Q is estimated as follows:

$$m_{\text{Activ}} = x \cdot (m_{\text{Electrode}} - m_{\text{Substrate}})$$

$$n_{\text{Li}} = m_{\text{Activ}} / M_{\text{Activ}} \quad [\text{mmol}]$$

$$Q = n_{\text{Li}} \cdot F \cdot z / 3600 \quad [\text{mAh}] \quad (z = 1, F = 96\,485 \text{ C/mol})$$

$$q_{\text{M}} = Q / m_{\text{Electrode}} \quad [\text{mAh/g}]$$

$$q_{\text{A}} = Q / m_{\text{Activ}} \quad [\text{mAh/g}]$$

$$q_{\text{F}} = Q / A \quad [\text{mAh/cm}^2]$$

For these calculations the following material data shall be given:

² Viledon[®] is the tradename of a product supplied by Freudenberg Nonwovens. This information is given for the convenience of users of this standard and does not constitute an endorsement by IEC of the product named. Equivalent products may be used if they can be shown to lead to the same results.

- a) mass of the electrode (mass of coated foil), $m_{\text{Electrode}}$;
- b) mass of the substrate (mass of uncoated foil), $m_{\text{Substrate}}$;
- c) stoichiometry/molar mass of the active material, M (can be proven by chemical analysis i.e. ICP-MS analysis);
- d) mass fraction of the active material in the electrode, x
- e) electrode area, A .

4.4 Preparation of the screw cell

The cell components are cleaned with ethanol and water in an ultrasonic bath and afterwards dried in a compartment dryer. The components are stored in the compartment dryer at 70 °C – 80 °C for at least 30 min. During such heat treatment of cell components the occasionally adsorbed water from surface of components will be removed.

The warmed-up components of the cell are mounted as shown in Clause A.1. Afterwards they are put into the glove box to assemble the electrochemical package under argon atmosphere. All materials under this section must be handled under argon atmosphere in a glove box. In the glove box the maximum O₂ content is 50 ppm and the maximum H₂O content is 10 ppm.

The cathode is placed inside the cell body and impregnated with LP40 electrolyte (5 drops, for cell area 1,27 cm² and cathode thickness of 50 μm).

The separator with thickness of 190 μm is punched out and 2 layers are placed onto the cathode. A defined amount of LP40 electrolyte (300 mg or 5 drops dispensed from micropipette per separator layer) is put on the separator.

The lithium anode is punched out and mechanically pressed onto a stainless steel or titanium spacer to minimize contact resistance. Afterwards it is put on the separator. By use of stainless steel spacers the corrosion free spacer operation should be proven after disassembling the cell. Stainless steel spacers should be replaced by titanium spacers if corrosion is observed.

Finally the cell body is equipped with the stainless steel spring ($k = 2,87$ N/mm) and a valid number of stainless steel spacers, and the cell is screwed under pressure.

Table 1 – Spring force and pressure

	Spring	Spring + 1 spacer	Spring + 2 spacer	Spring + 3 spacer
Spring force / N	14,87	19,23	23,59	27,95
Pressure / kNm ⁻²	117	151	186	220

A brief function test is performed by determining the cell voltage with a multimeter:

$$U = (3 \pm 0,5) \text{ V (specific value of the materials)} \rightarrow \text{correct}$$

$$U < 1,6 \text{ V} \rightarrow \text{fail}$$

In case the OCV of the cells with the same type of the cathode is between 1,6 and 2,5 V, such cells can be cycled for 5 to 10 times. If the discharge capacity of the electrode is <35 % of theoretical capacity Q (<0,35* Q , see 4.3) or strong degradation (>50 % after 10 cycles or >10 % / cycle after 3rd cycle) of capacity is observed the results should be disregarded and the sample preparation optimized.