



BSI Standards Publication

Nanomanufacturing — Key control characteristics

Part 4-1: Cathode nanomaterials for
nano-enabled electrical energy storage —
Electrochemical characterisation,
2-electrode cell method

National foreword

This Published Document is the UK implementation of IEC/TS 62607-4-1:2015. It supersedes PD IEC/TS 62607-4-1:2014, which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee NTI/1, Nanotechnologies.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

© The British Standards Institution 2015.

Published by BSI Standards Limited 2015

ISBN 978 0 580 89147 2

ICS 07.030

Compliance with a British Standard cannot confer immunity from legal obligations.

This Published Document was published under the authority of the Standards Policy and Strategy Committee on 30 September 2015.

Amendments/corrigenda issued since publication

Date	Text affected
-------------	----------------------



TECHNICAL SPECIFICATION



**Nanomanufacturing – Key control characteristics –
Part 4-1: Cathode nanomaterials for nano-enabled electrical energy storage –
Electrochemical characterisation, 2-electrode cell method**

INTERNATIONAL
ELECTROTECHNICAL
COMMISSION

ICS 07.030

ISBN 978-2-8322-2852-4

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

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	7
4.2.1 Cathode foil	7
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.....	9
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	10
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 LFP electrode	15
Figure A.1 – Components for the cell	12
Figure A.2 – Construction steps a to g.....	15
Figure A.3 – Open circuit voltage/potential time graph.....	15
Figure A.4 – Electrochemical impedance graph	16
Figure A.5 – Constant current / constant voltage – Charge-discharge cycle.....	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 nano-enabled electrical energy storage – Electrochemical characterisation, 2-electrode cell method**

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 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.

This second edition cancels and replaces the first edition published in 2014. This edition constitutes a technical revision.

Following discussions between IEC TC 113 and IEC TC 21/SC 21A: Secondary cells and batteries containing alkaline or other non-acid electrolytes, this edition includes the following significant technical changes with respect to the previous edition:

- a) The title of IEC 62607-4-1 has been modified.
- b) The scope has been revised to clarify that this Technical Specification deals with a standardized method for the determination of electrochemical properties of cathode nanomaterials of, for example, lithium-ion batteries utilizing lithium iron phosphate.
- c) In 3.1.1, the definition of "cathode nanomaterial" has been revised to be more specific.

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

Enquiry draft	Report on voting
113/238/DTS	113/261A/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 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 publication will remain unchanged until the stability date indicated on the IEC website 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.

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

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 regarded 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 part of IEC 62607 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 comparison of the results of different research groups.

This part of IEC 62607 introduces a 2-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 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 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 part of IEC 62607, 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 4-1: Cathode nanomaterials for nano-enabled electrical energy storage – Electrochemical characterisation, 2-electrode cell method

1 Scope

This part of IEC 62607 provides a standardized method for the determination of electrochemical properties of cathode nanomaterials of, for example, lithium-ion batteries utilizing lithium iron phosphate 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 part of IEC 62607 includes:

- definitions of terminology used in this part of IEC 62607,
- 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.

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 terms and definitions given in ISO/TS 80004-1 and the following apply.

3.1.1

cathode nanomaterial

material used as a cathode in nano-enabled energy storage devices 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, as LFP), a conducting phase (carbon black) and an organic binder (PVDF).

3.1.2

screw cell

cell providing the geometrical conditions in the 2-electrode arrangement