

TECHNICAL SPECIFICATION



**Nanomanufacturing – Key control characteristics –
Part 4-4: Nano-enabled electrical energy storage – Thermal characterization of
nanomaterials, nail penetration method**

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

**NANOMANUFACTURING –
KEY CONTROL CHARACTERISTICS –****Part 4-4: Nano-enabled electrical energy storage – Thermal
characterization of nanomaterials, nail penetration method**

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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 TS 62607-4-4, 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
113/306/DTS	113/329/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- withdrawn,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.

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A bilingual version of this publication may be issued at a later date.

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INTRODUCTION

Energy storage devices are becoming increasingly important for many applications such as consumer devices, electric vehicles and aircrafts. Energy storage devices with high performance and reliability are the key factors to earn the confidence of customers. Also, in smart grid and renewable energy applications, where energy efficiency and reliable power supplies are critical, an energy storage system is an essential device. There are many types of energy storage devices for various applications. Lithium-ion batteries are the most popular and promising energy storage devices for portable electronics, consumer electronics, military, electric vehicle and aerospace applications. It is a good test carrier for performance and reliability characteristics.

One of the characteristics that draws the attention of users is the thermal runaway behaviour when a short circuit occurs inside the energy storage devices due to a manufacturing process defect, improper operation or external shocks. The poor control of manufacturing process may cause the energy storage device's internal defects, such as particle impurity, defects of separator, burr of electrodes or a prominence of conductive arms. Energy storage devices operated under abnormal conditions, such as quick charging or piercing by external objects, may cause an internal short circuit. Large current generated short circuit will generate an abnormal exothermic reaction and a local temperature rise, but the temperature of the short circuit spot drops due to heat transfer. These effects cause the energy storage device's temperature to continue to rise rapidly. If it reaches thermal runaway temperature, it usually leads to fire and explosion of the energy storage devices. The event can result in damage to personnel and equipment. In the worst case scenario, this may hamper the development of such type of energy storage devices.

In order to prevent such a scenario, nanomaterial additives have been used to prevent thermal runaway to ensure the reliability and safety of energy storage devices. The nanomaterial additives may mix with active materials of electrodes, electrolyte, coated on the surface of electrodes or separator.

This document specifies general testing procedures and requirements for the assessment of thermal runaway performance and risk associated with the nano-enabled energy storage devices prepared by employing nanomaterial additives, and serves as the basis for further developing particular product specific standards. This method covers only large temperature rises in cell temperature caused by shorting of the anode and cathode. This method does not generally cover thermal runaway due to other causes such as high external temperature and is not a general method to prevent thermal runaway.

NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 4-4: Nano-enabled electrical energy storage – Thermal characterization of nanomaterials, nail penetration method

1 Scope

This part of IEC 62607, which is a Technical Specification, provides a measurement method for thermal runaway quality level test for nano-enabled energy storage devices. This method uses comparative measurement to enable a manufacturer to decide whether or not the nanomaterial additives used in energy storage devices are resilient against the thermal runaway caused by a faulty or accidental low resistance connection between two or several internal points depending on the number of stacking electrode layers of the test sample. The nanomaterial additives may mix with the materials of positive and negative electrodes, electrolyte, coated on electrodes or separator. This document includes definitions of terminology, test sample, puncture nail requirements, test procedures, data analysis and methods of interpretation of results and a case study.

This document does not apply directly to the safety testing for energy storage device products due to complex safety design schemes embedded in these products.

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 9001:2015, *Quality management systems – Requirements*

ISO 14001:2015, *Environmental management systems – Requirements with guidance for use*

ISO 26000:2010, *Guidance on social responsibility*

ISO/TS 80004-1:2015, *Nanotechnologies – Vocabulary – Part 1: Core terms*

ISO/TS 80004-2:2015, *Nanotechnologies – Vocabulary – Part 2: Nano-objects*

ISO/TS 80004-4:2011, *Nanotechnologies – Vocabulary – Part 4: Nanostructured materials*

IEC TS 80004-9:2016, *Nanotechnologies – Vocabulary – Part 9: Nano-enabled electro-technical products and systems*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 9001, ISO 14001, ISO 26000, the core terms of ISO/TS 80004-1, ISO/TS 80004-2, ISO/TS 80004-4, IEC TS 80004-9 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

nanoscale

length range approximately from 1 nm to 100 nm

[SOURCE: ISO/TS 80004-1:2015, 2.1]

3.2

nanomaterial

material with any external dimension in the nanoscale or having internal structure or surface structure in the nanoscale

[SOURCE: ISO/TS 80004-1:2015, 2.4]

3.3

nano-object

discrete piece of material with one, two or three external dimensions in the nanoscale

[SOURCE: ISO/TS 80004-1:2015, 2.5]

3.4

nanomaterial additive

nanomaterial, added in small quantities to a part of a device, to improve or otherwise modify one or more properties

3.5

nanocomposite material

multiphase material where one of the phases has one, two or three dimensions of less than nanoscale

3.6

nano-enabled

exhibiting function or performance only possible with nanotechnology

[SOURCE: ISO/TS 80004-1:2015, 2.15]

3.7

nano-enhanced

exhibiting function or performance intensified or improved by nanotechnology

[SOURCE: ISO/TS 80004-1:2015, 2.16]

3.8

open circuit voltage

difference in electrical potential voltage between the terminals of a cell or battery measured when the circuit is open (no-load condition) and no external current is flowing

[SOURCE: ISO 17546:2016, 3.26]

3.9

short circuit

circuit with the impedance of less than 1 mΩ applied between the output terminal and ground

[SOURCE: ISO 27027:2014, 3.13]

3.10**cell**

single energy or charge-storing unit within a pack of cells that form the energy storage device

3.11**capacity**

electric charge which a cell or battery can deliver under specified discharge conditions

Note 1 to entry: The SI unit for electric charge, or quantity of electricity, is the coulomb (1 C = 1 A·s) but in practice, capacity is usually expressed in ampere hours (Ah).

[SOURCE: IEC 60050-482:2004, 482-03-14]

3.12**separator**

non-conductive semi-permeable film or grid to separate two electrodes to prevent them from contacting each other and short-circuiting but which allows the passage of ions through it

3.13**SOC****state of charge**

status of available energy in the energy storage device

Note 1 to entry: SOC is usually expressed as a percentage.

4 Sample preparation

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4.1 General sample requirements

Testing samples shall be fully activated energy storage device cells. The shape (usually prismatic or cylindrical), the inner structures (usually stacked or rolled), anode/cathode materials, electrolyte and separator are based on manufacturers' design and requirements. In order to establish distinguishable nano-enabled or nano-enhanced function, two sets of samples should be prepared. Set one is without nanomaterial additives (reference sample), the other set is with nanomaterial additives (test sample). The minimum number of cells are 20 and 10, respectively, for appropriate statistical significance. The nanomaterial additives could be blended into anode/cathode materials, electrolyte and surface of the separator. Apart from nanomaterial additives, the two test samples should be the same (for example, size, assembly method and structure, capacity, materials, etc). The specifications of the test sample should be illustrated.

4.2 Pre-treatment

Pre-treatment should be performed for reference sample and test sample to select the qualified samples for thermal runaway test. The pre-treatment processes are as follows.

- 1) Prepare 20 reference samples.
- 2) Prepare 10 test samples.
- 3) Perform three charge/discharge cycles according to 1,0 °C or self-defined conditions, the temperature should be controlled at (25 ± 5) °C.
- 4) The sample should be fully charged (100 % SOC) and kept at (25 ± 5) °C for one hour. The mean, standard deviation and variation coefficient of the electric capacity can be calculated from 1,0 °C discharge curve. If the variation coefficient is larger than 3 %, the sample batch is not qualified for thermal runaway test. Another batch of samples should be selected for pre-treatment.
- 5) If electric capacity of the sample is outside the range of mean $\pm 1,5$ sigma, it is not qualified for thermal runaway test.

- 6) The total number of reference samples for thermal runaway test should be larger than 10; the total number of test samples for thermal runaway test should be larger than three.
- 7) Different manufacture (designed) nanomaterial additive samples should be pre-treated separately according to steps 2) to 5).

4.3 Puncture nail construction

Several important factors, such as nail diameter, material, cone angle, etc., will affect the test results. It is crucial to standardize the nail structures in order to have reliable quantitative data for comparison. The design is to embed a thermocouple inside a puncture nail in order to reliably record the temperature around the nail apex. Nail structures are described as follows.

- 1) The outer diameter of the nail is 2,5 mm, the tapered length is 2 mm, the cone angle is 64° , the inner diameter of the nail is 1 mm and the depth is equal to the cylindrical length (excluding taper).
- 2) The length of the nail (including taper) must be 10 mm longer than the energy storage device's height.
- 3) Thermocouple is embedded inside the nail inner cylinder (high temperature thermocouple is recommended). The inner wall of the nail is coated with thermal insulated magnesium oxide (MgO) to prevent measurement interference.
- 4) The puncture nail material is made of AISI 300 series stainless steel. The other parts connecting to the puncture nail should be mechanically strong, corrosion/fire resistant and easy to maintain.
- 5) The schematic view of the puncture nail is illustrated in Figure 1.

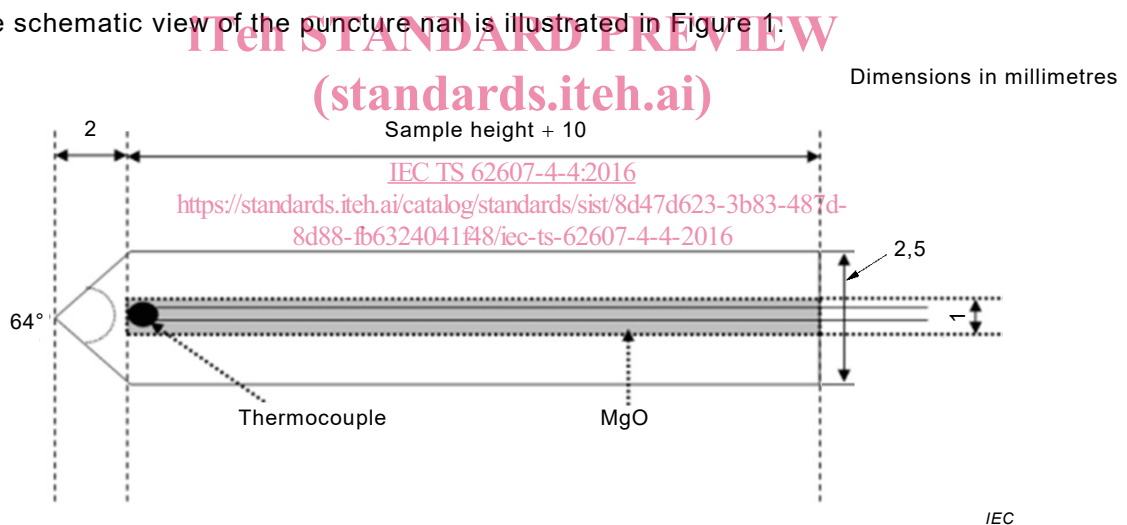


Figure 1 – Schematic view of puncture nail

4.4 Puncture nail temperature and isolation verification

In order to ensure the temperature reading at the puncture point and minimize the reading interference from non ideal MgO thermal isolation, the puncture nail temperature and isolation verification procedure need to be performed. The procedures are as follows.

- 1) Select a good thermal conducting metal block (thickness about 20 mm) with a hole at the top, the diameter of the hole is 2,5 mm with depth equal to 10 mm.
- 2) The metal block is heated to 160°C . Thermal equilibrium of the metal block is checked with two thermocouples, one is at the top and the other is at the side as shown in Figure 2. Insert the nail in to the hole of the metal block when thermal equilibrium of the metal block is reached. Wait until the reading of the thermocouple of the nail is stable.
- 3) The construction of the puncture nail is acceptable if the temperature reading of the nail is $\pm 3^\circ\text{C}$ at 160°C .