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TECHNICAL SPECIFICATION

colour inside

Nanomanufacturing – Key control characteristics – Part 4-4: Nano-enabled electrical energy storage – Thermal characterization of nanomaterials, nail penetration 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 62607-4-4, 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- withdrawn,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.

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://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
nano composite 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

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 ± 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 strutures 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.

Dimensions in millimetres

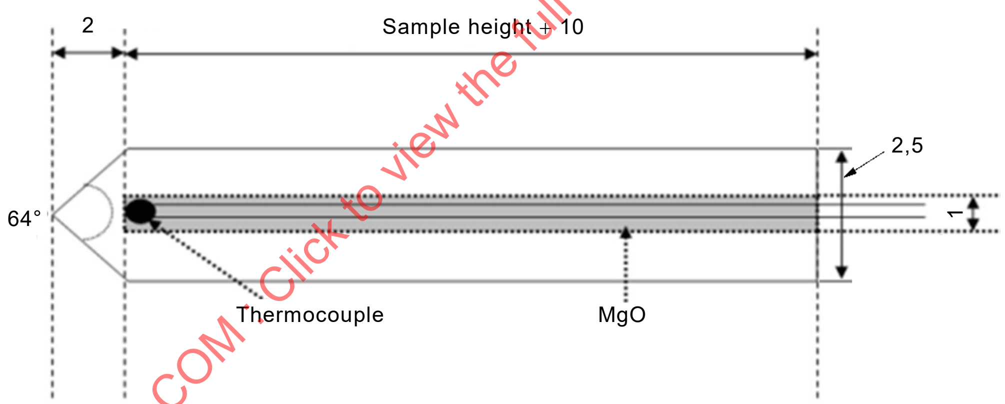

Figure 1 – Schematic view of puncture nail

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 ±3 °C at 160 °C.

4) The electric insulation is tested with a multimeter after the puncture nail has cooled to room temperature.
5) The schematic view of the puncture nail and the heated metal block is illustrated in Figure 2.

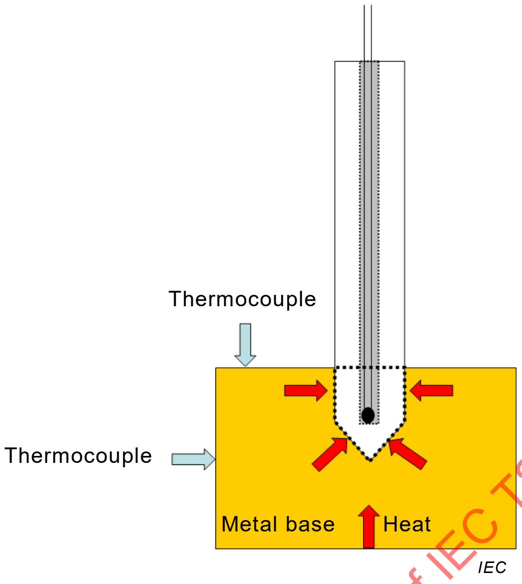

Figure 2 – Schemetic view of puncture nail and the heated metal block

5 Measurement of electric properties

5.1 Equipment and testing site requirements

The safety requirements for different samples are quite different. The measurement equipment should be able to be applied to various samples, yet it can achieve consistent accuracy. Generally, the equipment can provide adequate puncture force for different hardness samples. The measurement should also provide safe observation setup for dangerous results (such as smoke, fire and explosion, etc.). The equipment requirements and recommendations are as follows.

1) The maximum controlled displacement velocity of the servo motor should be equal to or larger than 50 mm/s, the minimum should be equal to or less than 0,1 mm/s, the error should be less than 5 %.
2) The puncture force shall be at least 5 000 N (It can puncture a soda metal can.).
3) The testing site shall be well sealed and the temperature shall be controlled in (25 ± 5) °C range.
4) The testing site should be smoke, fire and explosion resistant, with good ventilation and good pressure relief.
5) The testing site shall have an isolated observation window; a video camera or other image collection device can be used to record the event.

5.2 Measurement system requirements

The short circuit occurs very quickly. The voltage and temperature readings are very important to properly decide the results from these indicators. In order to record real time response of the sample, the measurement system shall satisfy the following requirements and recommendations.

1) The measurement system shall record voltage, temperature and nail position as a function of time.

2) Temperature and voltage sampling rate should be larger than 100 Hz (100 records per second).
3) Voltage sensitivity should be better than ±50 mV; the thermocouple can withstand temperatures higher than 1 000 °C.

5.3 Sample setup

There are various shapes and structures of the samples. The sample setup requirements are different for different shapes. A clear description of the sample setup is thus required. The following sessions describe the setup for general sample shapes, cylinder and square. The general principles can be applied to other shapes.

1) When punctured the sample shall not rotate, move or deform. The sample can be placed on the insulated and flame resistant surface. If needed (for cylindrical type cell), the sample can be fixed with fixture, but cannot affect the heat radiation and thermal expansion.
2) The heat shrink film, if any, is removed to prevent measurement interference.
3) A thermocouple is to be placed at the top surface of the sample. The position should be near the puncture point without affecting puncture procedure. The thermocouple should be fixed to avoid position shift due to smoke, fire or explosion.
4) The voltage signal is monitored by connecting to the terminals of the meter.

5.4 Puncture method

There are different kinds of sample assembly patterns, such as stacked and spiral. If the puncture depth is different, the damage inside the sample will be different. The measuring temperature point will also be different. These will cause inconsistent results. It is important to define the puncture direction and depth. The puncture method is as follows.

1) The nail puncture direction is perpendicular to the sample's electrode plane. The direction can be Z-axis (vertical) or XY-axis (horizontal). The puncture speed and force should follow the test specification described in 5.5 1).
2) The puncture through depth of cell is 4 mm, as shown in Figure 3.

Dimensions in millimetres

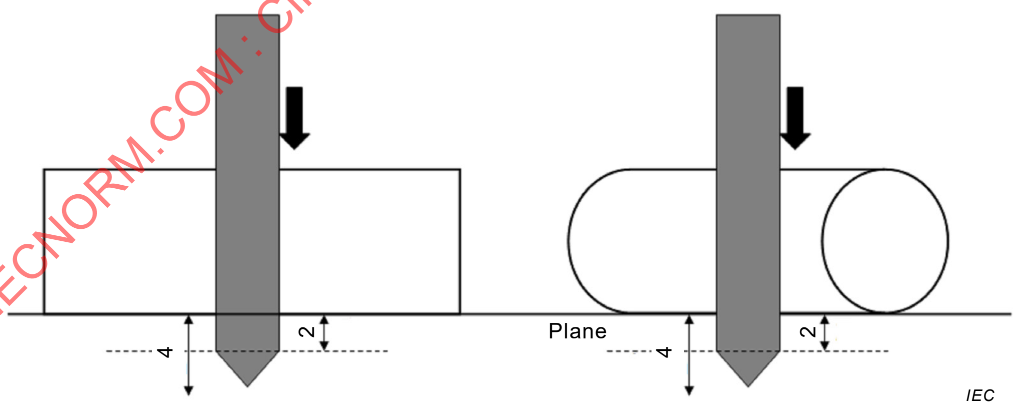

Figure 3 – The puncture through depth of cell

3) The recording of voltage and temperature is synchronized with the movement of the nail.
4) Once the temperature is stable, the sample can be removed. The puncture nail should not be reused and should be discarded.

5.5 Design of testing method

The testing method is a comparative methodology with two different samples, one is the reference sample (without nanomaterial), the other one is the test sample (with nanomaterial). The test procedures are as follows.

1) The puncture nail punctures the reference sample at several different speeds between 0,1 mm/s and 50 mm/s. The starting test speed is 25 mm/s, the following speed selection is based on high/low hazard levels dichotomy. Figure 4 shows the flow chart of test procedures. The high/low hazard levels are described in Table 1. The recommended number of tests is four to five for the reference sample and three to five for the test sample.
2) Repeat process 1) two to four times for repeatability test.
3) If the repeatability result fails, select another batch of reference samples and test again.
4) Temperature, voltage data and hazard level should be recorded.

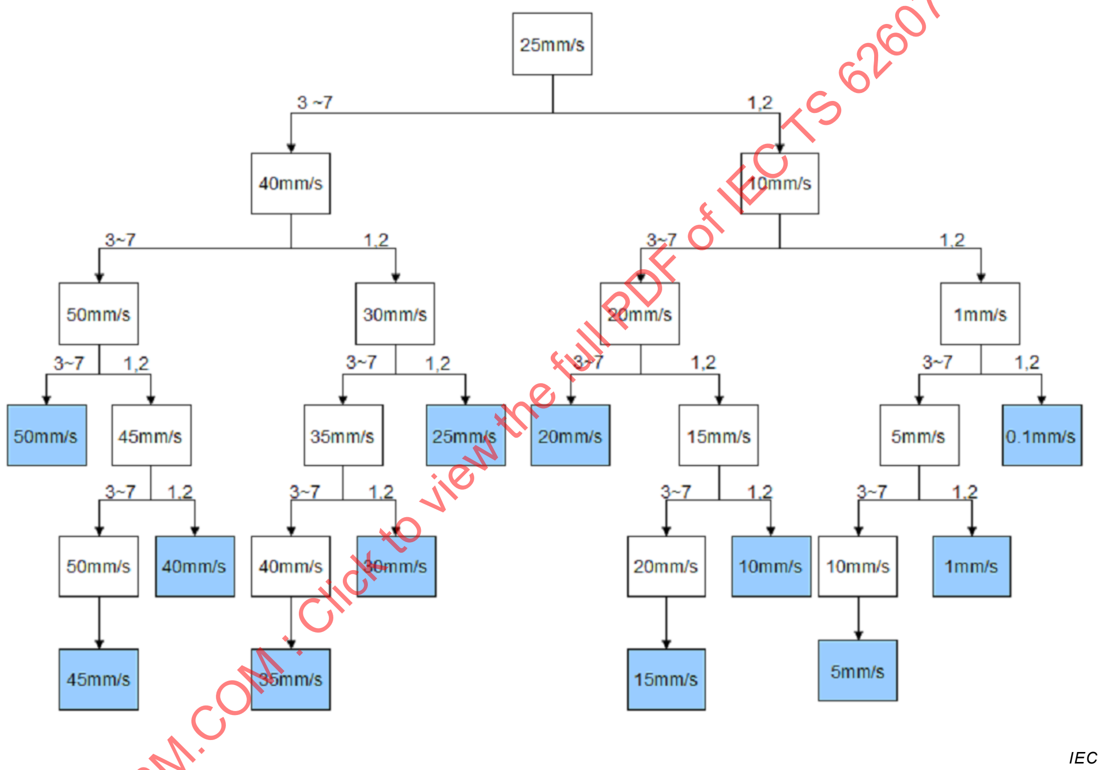

Figure 4 – Flow chart of reference sample test procedures

Table 1 – Hazard level description

Hazard level	Results	Abbreviation	Description
1	No smoke, no fire	P	No significant change in appearance
2	Vent	V	Electrolyte evaporation
3	Smoke	S	Swelling, the appearance of charred, emitting smoke
4	Spark + smoke	SS	Smoke and sparks
5	Heavy smoke	HS	A lot of smoke coming out
6	Fire	F	On fire
7	Explosion	EX	Explosion

6 Data analysis / interpretation of result

6.1 Samples classification from the results

6.1.1 Reference sample (without nanomaterials)

1) Results P and V are low temperature safe zone.
2) Results S, SS, HS and F are high temperature danger zone. HS and F results will cause the greatest hazard.

6.1.2 Test sample (with nanomaterials)

1) If the results of the reference samples and test samples are P or V, it indicates that the nanomaterial additive does not contain performance enabled or enhanced functionality.
2) If the results of the reference samples and test samples are S or SS or HS, it indicates that the nanomaterial additive does not contain performance enabled or enhanced functionality.
3) If the results of reference samples are S or SS or HS, and the results of test samples are F or EX, it indicates that the nanomaterial additive worsens the performance.
4) If the results of reference samples are S or SS or HS, and the results of test samples are P or V, it indicates that the nanomaterial additive does contain performance enabled or enhanced functionality.
5) If the results of reference samples are F or EX, and the results of test samples are S or SS or HS, it indicates that the nanomaterial additive contains limited performance enabled or enhanced functionality.
6) If the results of reference samples are F or EX, and the results of test samples are P or V, it indicates that the nanomaterial additive contains performance enabled or enhanced functionality.

6.2 Data analysis

1) Single test sample plot: to create a plot of voltage and temperatures versus time. The temperatures include surface and puncture point temperature. This plot can analyse voltage drop and temperature increment when puncture is initiated as shown in Figure 5.

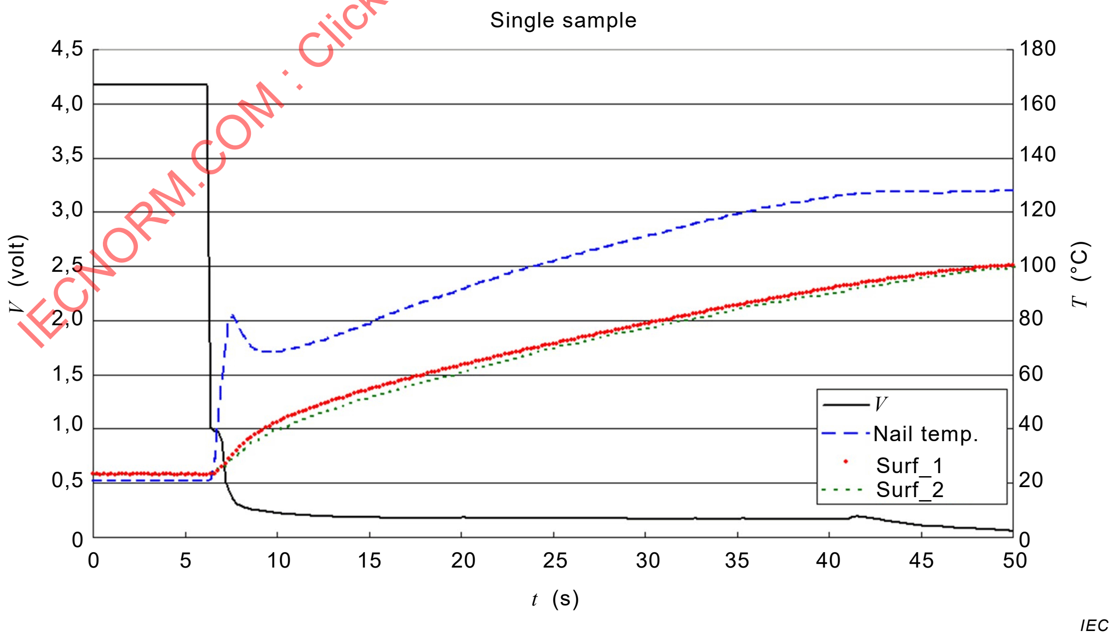

Figure 5 – Voltage and temperatures versus time plot

2) Repeatability plot: to create a plot of voltage and temperature versus time of multiple samples. The temperature includes only puncture point temperature. This plot can analyse whether the repeatability is good or not as shown in Figure 6.

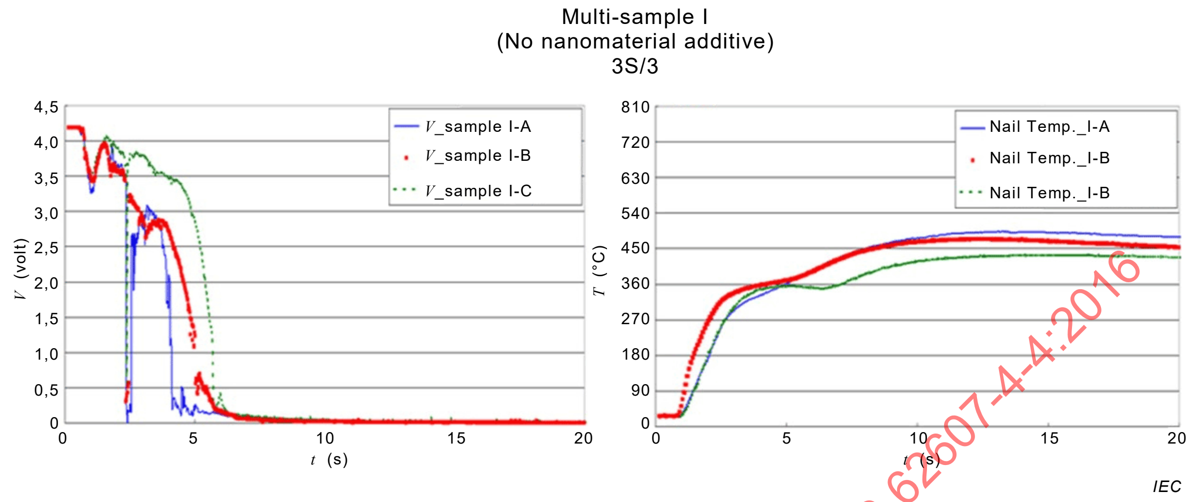

Figure 6 – Repeatability plot of voltage and temperature versus time

3) Performance comparison plot: to create a plot of voltage and temperature versus time of different types of nanomaterial additive samples. The temperature includes only puncture point temperature. Samples with different types of nanomaterial additive are plotted together for comparison. This plot can determine whether the performance is improved or not due to nanomaterial additive as shown in Figure 7. If the test results are difficult to discriminate, further quantitative analysis of nail temperature can discriminate the performance change.

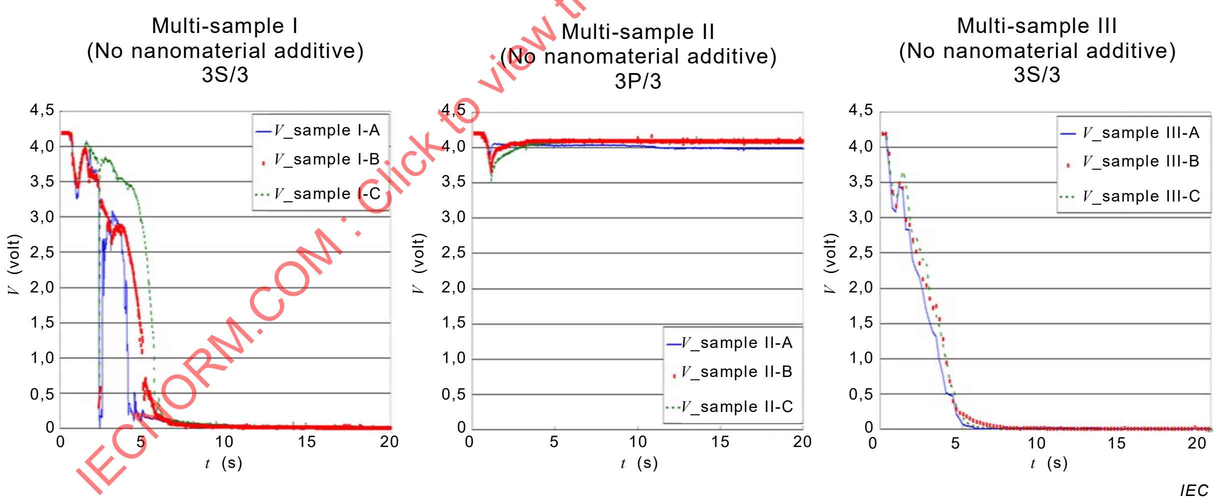

Figure 7 – Performance comparison plot of voltage and temperature versus time

Annex A
(informative)

Data report example

Tables A.1 to A.5 illustrate the specification table examples for anode, cathode, electrolyte, separator and cell. Table A.6 illustrates the sample analysis table for different additives.

Table A.1 – Anode specifications

Sample	Anode material	Energy density	Thickness of Al foil	Nanomaterial
1	same	same	same	X
2				P-1
3				P-2

Table A.2 – Cathode specifications

Sample	Cathode material	Energy density	Thickness of Cu foil	Nanomaterial
1	same	same	same	X
4				N-1
5				N-2

Table A.3 – Electrolyte specifications

Sample	Electrolyte formula	Nanomaterial
1	same	X
6		E-1
7		E-2

Table A.4 – Separator specifications

Sample	Material	Aperture	Shutdown temperature	Coating
1	same	same	same	X
8				C-1
9				C-2

Table A.5 – Cell specification

sample	Electrode structure	Container	Capacity	size
1	same	same	same	same
2				
3				
4				
5				
6				
7				
8				
9				