

TECHNICAL SPECIFICATION

**Nanomanufacturing – Material specifications –
Part 5-2: Nano-enabled electrodes of electrochemical capacitors – Blank detail
specification**

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INTERNATIONAL
ELECTROTECHNICAL
COMMISSION

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

NANOMANUFACTURING – MATERIAL SPECIFICATIONS –**Part 5-2: Nano-enabled electrodes of electrochemical capacitor –
Blank detail specification**

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IEC TS 62565-5-2 has been prepared by IEC technical committee 113: Nanotechnology for electrochemical products and systems. It is a Technical Specification.

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

Draft	Report on voting
113/628/DTS	113/643/RVDTS

Full information on the voting for its approval can be found in the report on voting indicated in the above table.

The language used for the development of this Technical Specification is English.

This document was drafted in accordance with ISO/IEC Directives, Part 2, and developed in accordance with ISO/IEC Directives, Part 1 and ISO/IEC Directives, IEC Supplement, available at www.iec.ch/members_experts/refdocs. The main document types developed by IEC are described in greater detail at www.iec.ch/standardsdev/publications.

A list of all parts in the IEC 62565 series, published under the general title *Nanomanufacturing – Material specifications*, 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 webstore.iec.ch in the data related to the specific document. At this date, the document will be

- reconfirmed,
- withdrawn,
- replaced by a revised edition, or
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INTRODUCTION

This Technical Specification specifies how to report the various characteristics of electrodes for industrial use in electrotechnical products, and how to incorporate these into a bilateral detail specification between vendor and user.

Electrochemical capacitors are widely used in the fields of electric vehicles, high speed trains, aircraft, photovoltaic, wind power and electronics, due to their ultra-fast charge/discharge capability, long cycle life, wide working temperature range, high security reliability and low maintenance cost [1]¹. In the manufacture process of electrochemical capacitor, the electrode is a bridge between raw material and device. Therefore, the performance of the electrode is very critical in the whole electrochemical capacitor industry chain because the properties of electrodes not only reflect the performance of upstream raw material but also determine the performance of electrochemical capacitor [2][3][4][5][6][7][8][9].

For the purposes of development and commercialization of raw nanomaterials for electrodes and the electrochemical capacitor and assembly produced therefrom, the product characteristics and characterization methods need to be specified in a standardized way. This blank detail specification will benefit different stakeholders as follows: for material suppliers, it provides necessary feedback from the manufacturers to guide the design and production of raw materials; for the end-product manufacturers, it provides a toolbox for evaluating product quality so as to manage and improve process control, yield of products; for commercialization and trade, it provides a guidance on referred test methods for electrode classification; in addition, it will strengthen the links between material manufacture and down-stream user.

In this blank detail specification, the key chemical, physical, structural and electrochemical characteristics that will significantly influence the performance of electrochemical capacitors and their measurement methods are listed. These characteristics and characterization methods are not limited only to nano-enabled electrodes but also can be reference for other electrodes which are constructed by coating electrode materials on a current collector.

¹ Numbers in square brackets refer to the Bibliography.

NANOMANUFACTURING – MATERIAL SPECIFICATIONS –

Part 5-2: Nano-enabled electrodes of electrochemical capacitor – Blank detail specification

1 Scope

This part of IEC 62565, which is a Technical Specification, establishes a blank detail specification that lists the relevant key control characteristics (KCC) including chemical, physical, structural, and electrochemical characteristics of nano-enabled electrode for electrochemical capacitors. Electrodes of both electric double layer capacitors and pseudo capacitors with nano/ nanostructured materials such as nanoporous activated carbon, graphene, carbon nanotube, carbon black, carbon aerogel, carbon nanomaterial coating collector, etc., are included. For other electrodes, this document can be used for reference.

In addition, this document enables the customer to specify requirements in a standardized manner and to verify through standardized methods that the nano-enabled electrode of the electrochemical capacitors meets the required properties.

Numeric values to be specified for the characteristics in this document are intentionally left blank and are determined by agreement between customer and electrochemical capacitor supplier. Properties and characteristics deemed by the customer or supplier as not relevant to a specific application are classified as "not applicable" or "not specified".

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.

IEC TS 62607-4-2, *Nanomanufacturing – Key control characteristics – Part 4-2: Nano-enabled electrical energy storage – Physical characterization of cathode nanomaterials, density measurement*

IEC TS 62607-4-3, *Nanomanufacturing – Key control characteristics – Part 4-3: Nano-enabled electrical energy storage – Contact and coating resistivity measurements for nanomaterials*

IEC TS 62607-4-8, *Nanomanufacturing – Key control characteristics – Part 4-8: Nano-enabled electrical energy storage – Determination of water content in electrode nanomaterials, Karl Fischer method*

IEC TS 62607-6-20, *Nanomanufacturing – Key control characteristics – Part 6-20: Graphene-based material – Metallic impurity content: ICP-MS²*

ISO 9277, *Determination of the specific surface area of solids by gas adsorption – BET method*

ISO 15901-2, *Pore size distribution and porosity of solid materials by mercury porosimetry and gas adsorption – Part 2: Analysis of nanopores by gas adsorption*

² Under preparation. Stage at the time of publication: IEC DTS 62607-6-20:2021.

ISO 25178 (all parts), *Geometrical product specifications (GPS) – Surface texture: Areal*

3 Terms and definitions

For the purposes of this document, the following terms and definitions 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 General terms

3.1.1

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, modified – The two notes have been removed.]

3.1.2

nanostuctured material

material having internal nanostructure or surface nanostructure

Note 1 to entry: This definition does not exclude the possibility for a nano-object to have internal structure or surface structure. If external dimension(s) are in the nanoscale, the term nano-object is recommended.

[SOURCE: ISO/TS 80004-1:2015, 2.7]

3.1.3

nano-enabled device

device in which the material elements or assembly of such elements exhibit performance or function only possible with nanotechnology

Note 1 to entry: The material element is a nanomaterial.

Note 2 to entry: The performance or function exhibited is measurable and significant for the application of the nano-enabled device.

Note 3 to entry: Applications of nano-enabled devices can include, but are not limited to, energy storage devices (capacitors, materials for lithium ion battery, fuel cell membrane, etc.), photovoltaic, organic electronics, and electro-optical devices.

[SOURCE: IEC 80004-9:2017, 3.1.5, modified – Notes 1 and 2 have been modified.]

3.1.4

key control characteristic

KCC

key performance indicator

material property or intermediate product characteristic which can affect safety or compliance with regulations, fit, function, performance, quality, reliability or subsequent processing of the final product

Note 1 to entry: The measurement of a key control characteristic is described in a standardized measurement procedure with known accuracy and precision.

Note 2 to entry: It is possible to define more than one measurement method for a key control characteristic if the correlation of the results is well-defined and known.

3.1.5 blank detail specification BDS

structured generic specification of the set of key control characteristics which are needed to describe a specific nano-enabled product without assigning specific values and/or attributes

Note 1 to entry: The templates defined in a blank detail specification list the key control characteristics for the nano-enabled material or product without assigning specific values to it.

Note 2 to entry: Examples of nano-enabled products are: nanomaterials, nanocomposites and nano-subassemblies.

Note 3 to entry: Blank detail specifications are intended to be used by industrial users to prepare their detail specifications used in bilateral procurement contracts. A blank detail specification facilitates the comparison and benchmarking of different materials. Furthermore, a standardized format makes procurement more efficient and more error robust.

3.2 Terms related to capacitors

3.2.1 electrochemical capacitor supercapacitor

device that stores electrical energy using a double layer in an electrochemical cell

Note 1 to entry: The electrochemical capacitor is not to be confused with electrolytic capacitors.

[SOURCE: IEC 60050-114:2014, 114-03-03]

3.2.2 electrode

conductive part in electric contact with a medium of lower conductivity and intended to perform one or more of the functions of emitting charge carriers to or receiving charge carriers from that medium or to establish an electric field in that medium

[SOURCE: IEC 60050-151:2001, 151-13-01]

3.2.3 electrode type

classification of the electrode of an electrochemical capacitor according to the charge-storage mechanism

Note 1 to entry: There are two electrochemical capacitor types: electric double layer capacitor and pseudo capacitor.

3.2.4 electric double layer capacitor EDLC

device for electrostatic storage of electrical energy achieved by separation of charge in a double layer

[SOURCE: ISO 18300:2016, 3.8]

3.2.5 pseudo capacitor Faraday capacitor

device for storage of electrical energy achieved by rapid oxidation-reduction reaction near the surface of electrode

3.3 General product description and procurement information

3.3.1

active material

material that can be used to store energy by electrochemical double-layer or pseudo capacitance effect

Note 1 to entry: Typically, nonreactive carbon materials are electric double-layer capacitance active material, including activated carbon, pure carbon nanotube and pure graphene.

Note 2 to entry: Typically, carbon composites and carbons embedded with heteroatoms are pseudo capacitance active material.

3.3.2

additive

material added in small quantities to a liquid or granular material to produce some desired modification to its characteristics

[SOURCE: ISO 6707-1:2020, 3.4.4.1]

3.3.3

nano-additive

additive that is nanomaterial or nano-structured material

3.3.4

graphene

graphene layer

single-layer graphene

monolayer graphene

1LG

single layer of carbon atoms with each atom bound to three neighbours in a honeycomb structure

Note 1 to entry: It is an important building block of many carbon nano-objects.

Note 2 to entry: As graphene is a single layer, it is also sometimes called monolayer graphene or single-layer graphene and abbreviated as 1LG to distinguish it from bilayer graphene (2LG) and few-layered graphene (FLG).

Note 3 to entry: Graphene has edges and can have defects and grain boundaries where the bonding is disrupted.

[SOURCE: ISO/TS 80004-13:2017, 3.1.2.1]

3.3.5

nanofibre

nano-object with two similar external dimensions in the nanoscale and the third dimension significantly larger

Note 1 to entry: The largest external dimension is not necessarily in the nanoscale.

Note 2 to entry: The terms nanofibril and nanofilament can also be used.

[SOURCE: ISO/TS 80004-2:2015, 4.5]

3.3.6

nanotube

hollow nanofibre

[SOURCE: ISO/TS 80004-2:2015, 4.8]

3.3.7

carbon nanotube

nanotube composed of carbon

[SOURCE: ISO/TS 80004-3:2020, 3.3.3, modified – Note 1 has been removed.]

3.3.8

carbon black

compounding ingredient consisting essentially of more than 95 % elemental carbon in the form of near-spherical particles with major diameters less than 1 µm, generally coalesced into aggregates

[SOURCE: ISO 1382:2020, 3.75, modified – Note 1 has been removed.]

3.3.9

current collector

conductive material in a fuel cell that collects electrons from the anode side or conducts electrons to the cathode side

[SOURCE: IEC 60050-485:2020, 485-06-07]

3.3.10

aluminium foil

sheet of aluminium metal, usually less than 150 µm thick, that may be laminated to other materials such as kraft paper or polyethylene

[SOURCE: ISO 9229:2020, 3.5.7]

3.3.11

carbonaceous coating aluminium foil

aluminium foil coated with carbon nanomaterial

3.4 Chemical key control characteristics

3.4.1

water content

ratio, expressed in percent, between the mass of water contained in the material as received and the corresponding dry residue of the material

[SOURCE: ISO/TS 21268-2:2019, 3.6]

3.4.2

ash content

percent by mass of carbon-free residue on combustion and pyrolysis

[SOURCE: ISO 1998-2:1998, 2.10.120]

3.4.3

metallic impurities

metallic elements, such as Fe, Co, Ni, present but not intentionally added to a material, and the minimum content of which is not controlled

3.5 Physical key control characteristics

3.5.1

bending strength

maximum bending radius of the device or assembly at which the coating layer of electrode begins to crack or exfoliate from the current collector after applying the electrode to a film-bending tester

3.5.2**peel strength**

force per unit effective width required to separate the electrode materials from the substrate current collector under the specified conditions of test

3.5.3**rebound rate**

after calendaring, thickness change ratio of the electrode at different time points

3.5.4**volume resistivity** ρ_V

measured volume resistance calculated to apply to a cube of unit side

Note 1 to entry: It is expressed in ohm metres ($\Omega \cdot m$).

[SOURCE: ISO 14309:2019, 3.3]

3.5.5**electrolyte adsorption capacity**

ability of electrode to adsorb electrolyte, which can be determined by the time when a certain amount of electrolyte is absorbed by electrode with a certain area.

3.6 Structural key control characteristics**3.6.1****thickness**

shortest distance between two surfaces limiting a layer, when this distance can be considered to be a constant over a region of a finite size

[SOURCE IEC 60050-113:2011, 113-01-24]

3.6.2**surface density****areic mass****surface mass density**

at a given point on a two-dimensional domain of quasi-infinitesimal area dA , scalar quantity equal to the mass dm within the domain divided by the area dA , thus $\rho_A = dm/dA$

[SOURCE: IEC 60050-113:2011, 113-03-10, modified – The note has been removed.]

3.6.3**rolling density**

ratio of the mass of the rolled active material to its volume after being coated on a substrate

[SOURCE: IEC TS 62607-4-2, 3.1.3]

3.6.4**specific surface area**

absolute surface area of the sample divided by sample mass

[SOURCE: ISO/TS 11931:2012, 3.3]

3.6.5**pore volume**

volume of open pores unless otherwise stated

[SOURCE: ISO 15901-1:2016, 3.14]

3.6.6

surface roughness

surface topology of an area of surface, qualified in the International Standards by reference to various resolution determinants and methods of calculation

[SOURCE ISO/IEC 10373-2:2015, 3.7]

3.7 Electrochemical key control characteristics

3.7.1

capacitance

ability of a capacitor to store electrical charge

Note 1 to entry: Unit: farad (F).

[SOURCE: IEC 62576:2018, 3.5]

3.7.2

specific capacitance of electrode

capacitance of capacitor divided by the mass or volume of electrode

Note 1 to entry: Unit: farad per gram (F/g) or farad per cubic centimetre (F/cm³).

3.7.3

leakage current

value of the current that flows through a capacitor after a charge for a fixed period of time

Note 1 to entry: Leakage current is given in amperes (A).

Note 2 to entry: Leakage current is caused by an unwanted conductive path other than a short circuit.

[SOURCE: IEC 62391-1:2015, 3.28, modified – Note 2 to entry has been added.]

3.7.4

voltage maintenance rate ratio of voltage maintenance

ratio of the voltage at the open-ended terminals to the charge voltage after a specified time period subsequent to the charging of a capacitor

[SOURCE: IEC 62576:2018, 3.25]

3.7.5

endurance in cycling

number of charge and discharge cycles when the measured capacitance or internal resistance value reaches a specified degree of its initial value under a certain temperature and a certain rate of charge current

3.7.6

temperature endurance

ratio of the capacitance or internal resistance to its initial value after a specified charging time at constant voltage under a specific temperature

3.7.7

internal resistance

combined resistance of constituent material specific resistance and inside connection resistance of a capacitor

Note 1 to entry: Unit: ohm (Ω).

[SOURCE: IEC 62576:2018, 3.15]

3.8 Measurement methods relevant for this document

3.8.1

inductively coupled plasma mass spectroscopy

ICP-MS

analytical method for qualitative determination and quantitative evaluation of element concentrations by measuring the ions produced by a radiofrequency inductively coupled plasma

Note 1 to entry: In the mass spectrometer the ions are separated and the elements identified according to their mass-to-charge ratio m/z , while the concentration of the elements is proportional to the numbers of ions.

[SOURCE: ISO 7086-1:2019, 3.5]

3.8.2

inductively coupled plasma optical emission spectroscopy

ICP-OES

trace-level, elemental analysis technique that uses the emission spectra of a sample to identify and quantify the elements present

[SOURCE: ISO 7086-1:2019, 3.6]

3.8.3

atomic absorption spectrometry

AAS

spectroanalytical method for qualitative determination and quantitative evaluation of element concentrations wherein the technique determines these concentrations by measuring the atomic absorption of free atoms

[ISO 7086-1:2019, 3.2]

3.8.4

thermal gravimetric analysis

TGA

method in which the change in the mass of a sample is measured as a function of temperature while the sample is subjected to a controlled temperature programme

[SOURCE: ISO/TS 80004-6:2021, 6.1.2]

3.8.5

constant current discharge

CCD

discharge during which the electric current is maintained at a constant value regardless of the battery voltage or temperature

3.8.6

constant current charge

CCC

charge during which the electric current is maintained at a constant value regardless of the battery voltage or temperature

[SOURCE: IEC 60050-482:2004, 482-05-38]

3.8.7

constant voltage charge

CVC

charge during which the voltage is maintained at a constant value regardless of charge current or temperature

[SOURCE: IEC 60050-482:2004, 482-05-49]

4 General introduction regarding measurement methods

For reasons of practicality for industrial use in manufacturing of nano-enabled electrotechnical products, this document recommends appropriate measurement methods for each material parameter. The specification of some KCCs of nano-enabled electrodes of electrochemical capacitors refer to measurement procedures for which

- no standards currently exist, or
- standards are under development but have not yet been published, or
- standards were developed for other use cases but can be adopted with modifications.

In other cases, industrial users of this document shall fall back to methods which are used in the scientific community. As they are not established as documented measurement procedures, users of this document shall agree bilaterally on it. For the KCCs in this document, Clause 6 provides a general overview.

To fulfil the requirements of quality assessment management strategies this requires careful documentation of the measurement procedure. The document describing the method used shall have an identification number and include the following topics:

- a) Measurement principle:
 - basic scientific background;
 - measurement configuration if there are different experimental setups available which perform from a physical point of view the same measurement;
 - measurement mode if there are measurement modes possible with the experimental setup which deliver different kinds of information.
- b) Measurement system:
 - measurement equipment / apparatus;
 - materials;
 - calibration standards;
 - ambient conditions;
 - sample preparation method.
- c) Measurement procedure:
 - calibration of measurement equipment;
 - detailed protocol of the measurement procedure;
 - measurement accuracy.
- d) Data analysis / interpretation of results:
 - description of the method to derive the key control characteristics out of the measurement data including the used key formulas.
- e) Results to be reported:
 - description of the test sample including a sketch, drawing or photograph;
 - identification of the test sample, e.g. batch or serial number;
 - quantitative description of the accuracy of the measurement;

- measured key control characteristics listed in tables, plotted in figures, maps created by scanning methods.

As this document is intended to be updated regularly, there will be a standardized method for each KCC in the future, including supplementing, revising and replacing the reference standards. Nevertheless, for special applications supplier and customer may deviate from the recommendations and agree on other than the recommended standards or define a specific method for their application.

5 Specification format of nano-enabled electrode of electrochemical capacitor

5.1 General procurement information

General procurement information about nano-enabled electrode for electrochemical capacitors as well as a supplier-neutral technical description to classify the product should be provided by the manufacturer or product supplier according to Table 1.

Table 1 – Format for general product description and procurement information

Item No	ITEM	INFORMATION
1.1	Supplier	
1.2	Product	
1.3	Trade name	
1.4	Electrode type	
1.5	Active material	
1.6	ID number	
1.7	Description of the manufacturing process available	<input type="checkbox"/> No
		<input type="checkbox"/> Yes Reference
1.8	Typical batch quantity	<input type="checkbox"/> Mass [kg]
		<input type="checkbox"/> Volume [l]
1.9	Traceability requirements	<input type="checkbox"/> Batch number
		<input type="checkbox"/> Serial number
		<input type="checkbox"/> Others, specify
		Manufacturing date
1.10	Specification	Number
		Revision level
		Date of issue
1.11	Packaging requirements	
1.12	Material Safety Data Sheet (MSDS) available	<input type="checkbox"/> No
		<input type="checkbox"/> Yes Reference

NOTE Nanotechnology-based manufacturing processes are sometimes not mature enough that it can be excluded that there are hidden parameters with significant influence on the product performance. Therefore, a detailed description of the manufacturing process will help to control those parameters.

5.2 Chemical key control characteristics

Chemical characteristics as detailed in Table 2 shall be agreed between manufacturer and user. Characteristics not of relevance for the application may be classified as not applicable or not specified.

Table 2 – Format for chemical key control characteristics

ITEM NO.	KCC	SPECIFICATION	MEASUREMENT METHOD	MEASUREMENT PROCEDURE
2.1	Water content	Less than [] %	Karl Fischer method	IEC TS 62607-4-8 (Clause A.1)
2.2	Ash content	Less than [] %	Incineration	Not available (Clause A.2)
			Thermal gravimetric analysis (TGA)	Not available (Clause A.3)
2.3	Magnetic impurities content	Fe: Less than [] ppm Co: Less than [] ppm Ni: Less than [] ppm	ICP-MS	IEC TS 62607-6-20 (Clause A.4)
			ICP-OES	Not available (Clause A.5)
			AAS	Not available (Clause A.6)

5.3 Physical key control characteristics

Physical characteristics as detailed in Table 3 shall be agreed between manufacturer and user. Characteristics not of relevance for the application may be classified as not applicable or not specified.

Table 3 – Format for physical key control characteristics

ITEM NO.	KCC	SPECIFICATION	MEASUREMENT METHOD	MEASUREMENT PROCEDURE
3.1	Bending strength	Nominal [] ± Tolerance [] mm	Film bending tester	Not available (Clause A.7)
3.2	Peel strength	Nominal [] ± Tolerance [] N/m	Peel method	Not available (Clause A.8)
3.3	Rebound rate	Nominal [] ± Tolerance [] mm	Thickness variation	Not available (Clause A.9)
3.5	Electrolyte adsorption capacity	Nominal [] ± Tolerance [] S/mL	Time measurement	Not available (Clause A.10)
3.6	Contact angle	Nominal [] ± Tolerance [] °	Image method	Not available (Clause A.11)
3.7	Resistivity	Nominal [] ± Tolerance [] Ω·cm	Four-point probe resistivity tester	IEC TS 62607-4-3 (Clause A.12)

5.4 Structural key control characteristics

Structural characteristics as detailed in Table 4 shall be agreed between manufacturer and user. Characteristics not of relevance for the application may be classified as not applicable or not specified.

Table 4 – Format for structural key control characteristics

ITEM NO:	KCC	SPECIFICATION	MEASUREMENT METHOD	MEASUREMENT PROCEDURE
4.1	Rolling density	Nominal [] ± Tolerance [] g/cm ³	Weighing	IEC TS 62607-4-2 (Clause A.14)
4.2	Thickness	Nominal [] ± Tolerance [] mm	Micrometer	IEC TS 62607-4-2 (Clause A.13)
4.3	Surface density	Nominal [] ± Tolerance [] g/cm ²	Weighing	IEC TS 62607-4-2 (Clause A.14)
4.4	Specific surface area	Nominal [] ± Tolerance [] m ² /g	N ₂ adsorption	ISO 9277 (Clause A.15)
4.5	Pore volume	Nominal [] ± Tolerance [] cm ³ /g	Mercury porosimetry	ISO 15901-2 (Clause A.15)
4.6	Surface roughness	Nominal [] ± Tolerance [] nm	Optical microscope	ISO 25178 (Clause A.16)

5.5 Electrochemical key control characteristics

Electrochemical characteristics of electrodes as detailed in Table 5 shall be agreed between manufacturer and user. Characteristics not of relevance for the application may be classified as not applicable or not specified.

Table 5 – Format for electrochemical key control characteristics

ITEM NO.	KCC	SPECIFICATION	MEASUREMENT METHOD	MEASUREMENT PROCEDURE
5.1	Specific capacitance	Greater than [] F/g	Constant current charge (CCC) and constant current discharge (CCD)	Not available (Clause A.17)
5.2	Voltage maintenance rate	Greater than [] %	Constant current charge (CCC) and constant voltage charge (CVC)	Not available (Clause A.18)
5.3	Leakage current	Less than [] mA	Constant current charge (CCC) and constant voltage charge (CVC)	Not available (Clause A.19)
5.4	Endurance in cycling	Greater than [] % after n cycles	Constant current charge (CCC) and constant current discharge (CCD)	Not available (Clause A.20)
5.5	Temperature endurance	Greater than [] % after [] hours at [] °C	Constant voltage charge (CVC)	Not available (Clause A.21)
5.6	DC resistance	Less than [] Ω	Constant current charge (CCC) and constant current discharge (CCD)	Not available (Clause A.22)

6 Overview of test methods

For the entry under "measurement procedure" in the KCC tables, there are four scenarios regarding the availability of documented measurement methods, which are summarized in Table 6 for each combination of KCC and measurement method.

- Case 1) A standardized measurement method is not yet available, but the technical community has consensus about the need to specify the KCC. Also, a GPG is not available. This is the lowest level of common understanding in the community, and it is left to the parties involved in the delivery process to define a way of dealing with the situation, for example by adding an agreed standard operation procedure (SOP) to the specification. That shall be mentioned in Annex A also.
- Case 2) A standardized measurement method is not yet available, but the technical community has consensus about the need to specify the KCC. In this case, a Good Practice Guide (GPG) developed by a group of stakeholders or a consortium may serve as the basis for the measurement. The GPGs must be attached to the BDS as clauses in Annex B with an introduction of their use and a comment of their scientific validation. If the GPG is publicly available, it can be referenced instead.
- Case 3) A standardized measurement method is available which is intended to be used for another use case but can be adapted for the desired use case, e.g. other materials or other applications. They may not yet be validated for the use case in the BDS. In this case the method shall be listed in Annex A with a description of how the standard shall be adopted. Reference to the Annex A clause shall be given in the KCC tables.
- Case 4) A standardized measurement method is available and can be used exactly for the use case under consideration. In this case it is suitable just to list the standard in column "measurement procedure" of the KCC tables.

NOTE In Cases 1) and 2) it is suggested to transform the measurement protocol into a documented standard and to perform all necessary steps to prepare issuing a New Work Item Proposal to the IEC through the appropriate National Committee.

Annex A (normative)

KCC measurement procedures – supporting information

A.1 Water content: Karl Fischer method

A.1.1 General

Water content has a crucial impact for electrochemical capacitors. Residual water will adversely affect the resistance of the electrode and the stability and safety of the device. So it is important to restrict water content. In order to accurately measure the water content of the electrode, the Karl Fischer coulometric titration method should be used.

In the Karl Fischer coulometric titration method, first, iodine in Karl Fischer reagent is produced by electrolysis of the reagent containing iodide ion, and then the water content in a sample is determined by measuring the quantity of electricity which is required for the electrolysis (i.e. for production of iodine), based on the quantitative reaction of the generated iodine with water.

A.1.2 Documented measurement procedure

IEC TS 62607-4-8 is directly applicable to measure the water content.

A.2 Ash content: Incineration

A.2.1 General

Because of the raw material, reagent, equipment and environment in the fabrication process, some impurities will be present in the electrode. The existence of impurities, especially the magnetic Fe and Ni, adversely influences the performance of electrochemical capacitors significantly. Ash content can quickly reflect the impurities and is the percent by mass of carbon-free residue on combustion and pyrolysis at (800 ± 50) °C. The ash content of electrode can be determined by subtracting the incineration residue of the current collector from the incineration residue of the electrode.

A.2.2 Documented measurement procedure

A documented measurement procedure is not yet available (Case 1) and shall be agreed between customer and supplier.

A.3 Ash content: Thermal gravimetric analysis (TGA)

A.3.1 General

Because of the raw material, reagent, equipment and environment in the fabrication process, some impurities will be present in the electrode. The existence of impurities, especially the magnetic Fe and Ni, adversely influences the performance of electrochemical capacitors significantly. Ash content can quickly reflect the impurities and is the mass fraction, in per cent, of carbon-free residue on combustion and pyrolysis.

A.3.2 Documented measurement procedure

A documented measurement procedure is not yet available (Case 1) and have to be agreed between customer and supplier.

A.4 Magnetic impurities: ICP-MS

A.4.1 General

The presence of magnetic impurities can lead to an increase of self-discharge for electrochemical capacitor. Therefore, it is important to decrease the amount of magnetic impurities, such as Fe, Co, Ni. The magnetic impurities determination should be carried out through ICP-MS with the microwave digestion for pre-treatment.

A.4.2 Documented measurement procedure

IEC TS 62607-6-20 is applicable to measure the metallic impurity ash content. Even if IEC TS 62607-6-20 was originally developed for graphene-based materials, it can be used with care also for other materials covered by this document.

A.4.3 Adaptations required

a) Sample preparation

The sample shall undergo a pre-treatment by microwave digestion. Before the digestion, carefully scrape off the electrode material from the current collector. Then mix a specific mass of electrode material with some HNO₃ and digest.

A.5 Magnetic impurities: ICP-OES

A.5.1 General

The presence of magnetic impurities can lead to an increase of self-discharge for electrochemical capacitor. Therefore, it is important to determine the amount of magnetic impurities, such as Fe, Co, Ni. The magnetic impurities determination should be carried out through ICP-OES with the microwave digestion for pre-treatment. The sample preparation is same as in A.4.3 a).

A.5.2 Documented measurement procedure

A documented measurement procedure is not yet available (Case 1) and shall be agreed between customer and supplier.

A.6 Magnetic impurities: ASS

A.6.1 General

The presence of magnetic impurities can lead to an increase of self-discharge for electrochemical capacitor. Therefore, it is important to decrease the amount of magnetic impurities, such as Fe, Co, Ni. The magnetic impurities determination should be carried out through AAS with the microwave digestion for pre-treatment. The sample preparation is same as in A.4.3 a).

A.6.2 Documented measurement procedure

A documented measurement procedure is not yet available (Case 1) and shall be agreed between customer and supplier.

A.7 Bending strength: Lacquer cylinder bending tester

A.7.1 General

Bending strength reflects the mechanical strength of the electrode. The electrode bending strength is measured by using a lacquer cylinder bending tester. A series of electrodes are cut before winding (for example, dimension: length × width = 5 cm × 3,5 cm). The bending strength is measured from the largest diameter to the smallest until the electrode has a crack. The diameter at the same time that the electrode cracks is recorded and calculated as the bending strength of the electrode.

A.7.2 Documented measurement procedure

A documented measurement procedure is not yet available (Case 1) and shall be agreed between customer and supplier.

A.8 Peel strength

A.8.1 General

Peel strength is related to many performance characteristics of electrodes, such as cell self-discharge, cycle retention, electrode swelling during cycle and so on. Measurement of the peel strength of electrode is carried out using a universal testing machine. Cut the electrode into a rectangular piece with a certain length and width. Stick double-sided adhesive tape on the electrode surface, and stick the other side on a stainless-steel plate. The stainless-steel plate and collector are secured on two fixtures of the test equipment, and then a 180-degree peel test is carried out at a specified speed with a certain load. When the current collector is completely stripped off, the force detected is the stripping force.

A.8.2 Documented measurement procedure

A documented measurement procedure is not yet available (Case 1) and shall be agreed between customer and supplier.

A.9 Rebound rate

A.9.1 General

Electrode rebound is closely related to the structure of raw material and electrode processing. It can reflect the batch stability of material and stability of process. Generally, rebound is measured by recording the thickness of the electrode at different times after rolling. The rebound ratio is equal to the thickness at different times divided by the initial thickness.

A.9.2 Documented measurement procedure

A documented measurement procedure is not yet available (Case 1) and shall be agreed between customer and supplier.

A.10 Electrolyte adsorption capacity

A.10.1 General

Drop a certain volume of electrolyte on a certain area of electrode. The time from the electrolyte contacting the electrode to being completely absorbed divided by the volume of the electrolyte reflects the electrolyte adsorption capacity.

A.10.2 Documented measurement procedure

A documented measurement procedure is not yet available (Case 1) and shall be agreed between customer and supplier.

A.11 Contact angle

A.11.1 General

Drop a droplet of water or electrolyte on the surface of an electrode. Use a digital image capturing system to capture 50 frames per second. The contact angle is determined using the circle algorithm from digitized optical images.

A.11.2 Documented measurement procedure

A documented measurement procedure is not yet available (Case 1) and shall be agreed between customer and supplier.

A.12 Resistivity

A.12.1 General

Resistivity reflects the conducting capability of an electrode. It is related to the composition, structure and uniformity of electrode material. The electrode is prepared by coating the electrode slurry on a current collector with good conductivity, the current collector will severely affect the determination of the resistivity of the electrode. Therefore, it is important to determine the coating resistivity and the contact resistivity.

A.12.2 Documented measurement procedure

IEC TS 62607-4-3 is applicable to measure the resistivity. Even if IEC TS 62607-4-3 was originally developed for nanomaterials powder, it can be used with care also for other materials covered by this document.

A.12.3 Adaptations required

a) Sample preparation

In order to evaluate the resistivity of an electrode, the electrode slurry is coated on an isolator substrate rather than the current collector under the same preparation conditions as the tested electrode.

A.13 Thickness

Measuring the thickness can reflect the morphology and tap density of raw materials of electrodes. The thickness of an electrode is easily measured by using a micrometer. Measure several thickness values of the electrode from left side to right side. Take the arithmetical average of these values as the final thickness.

A.14 Surface and rolling density

A.14.1 General

Surface and rolling density can be measured by weighing method. Both electrode and current collector are punched into several small discs. Then use the electric balance to weigh and record the mass of discs one by one. Surface density is the average mass of the electrode