IEC TS 62565-5-1

Edition 1.0 2023-06

TECHNICAL SPECIFICATION

colour inside

Nanomanufacturing – Product specifications – Part 5-1: Nanoporous activated carbon – Blank detail specification: Electrochemical capacitors

IEC TS 62565-5-1:2023-06(en)

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

ICS 07.120

ISBN 978-2-8322-7096-7

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NANOMANUFACTURING – PRODUCT SPECIFICATIONS –

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FOREWORD

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IEC TS 62565-5-1 has been prepared by subcommittee 113: Nanotechnology for electrotechnical products and systems. It is a Technical Specification.

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

Draft	Report on voting
113/715/DTS	113/742/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/publications.

A list of all parts in the IEC 62565 series, published under the general title *Nanomanufacturing – Product specifications*, can be found on the IEC website.

Future documents in this series will carry the new general title as cited above. Titles of existing documents in this series will be updated at the time of the next edition.

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- withdrawn,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
- amended.

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INTRODUCTION

This document specifies how to report the various characteristics of nanoporous activated carbon for electrochemical capacitors, 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, airplanes, photovoltaics, wind power and electronics, due to their ultra-fast charge and discharge capability, long cycle life, wide working temperature range, high security reliability and low maintenance cost [1] [1]. Nanoporous activated carbon is the active material in electrochemical capacitors [2], [3], [4] (Figure 1), and is one of the most critical factors that determine the electrochemical performance of a device.

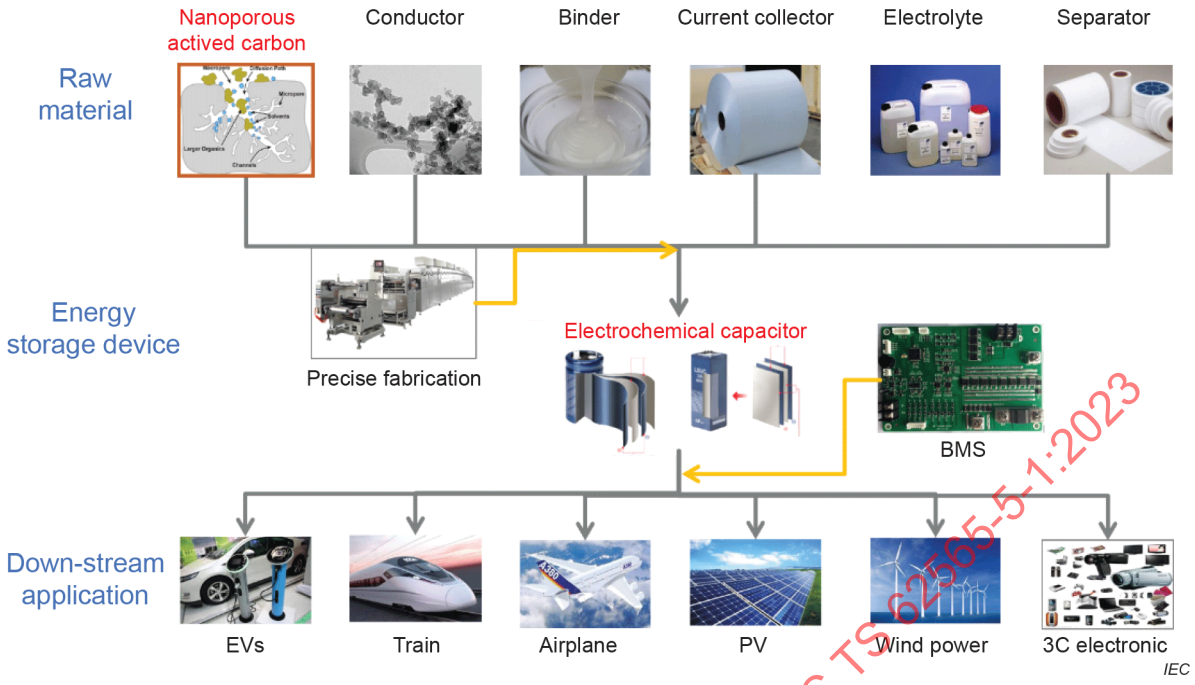
Both precursor and process will affect the chemical, physical and structural characteristics of nanoporous activated carbon remarkably. The precursor of nanoporous activated carbon can be biomass, pitch and resin. The production process can be gas activation using diluted oxygen gas, steam, CO_2, etc., or chemical activation using H_3PO_4, $ZnCl_2$, KOH, etc. The chemical, physical and structural key control characteristics (KCCs) will significantly affect the electrochemical performance of nanoporous activated carbon. For instance, the metallic impurities will affect the self-discharging and endurance in cycling, the pore size distribution will affect the specific capacitance and the DC resistance. However, not all relationships between the chemical, physical, structure and application properties of active materials are clear so far. In the commercial market, the KCCs will be good indicators to choose an appropriate nanoporous activated carbon. Therefore, it is important to report KCCs, including electrochemical characteristics.

For nanoporous activated carbon manufacturers, the accurate characterization is critical for product optimization, finalization and quality control. For electrochemical capacitor manufacturers, who use the nanoporous activated carbon, before the large-scale production of electrochemical capacitors, the correct and accurate characterization of KCCs can be good indicators for choosing the appropriate raw materials and achieving quality assurance

To permit common processing equipment and common unit processes with predictable and reproducible results to be used in different fabrication lines, it is important for nanoporous activated carbon characteristics to be described and assessed in a proper manner and to standardize the methods for quality control of the manufacturing processes.

In this document, the key chemical, physical, structural and electrochemical characteristics that will significantly influence the performance of electrochemical capacitors are listed. This document also provides information about measurement methods and existing standards concerning the correct determination of KCCs.

[1] Numbers in square brackets refer to the Bibliography.



Key

BMS battery management system

EVs electrical vehicles

PV photovoltaic power

3C computer, communication and consumer electronics

Figure 1 – Industrial chain of electrochemical capacitor

NANOMANUFACTURING – PRODUCT SPECIFICATIONS –

Part 5-1: Nanoporous activated carbon – Blank detail specification: Electrochemical capacitors

1 Scope

This part of IEC 62565, which is a Technical Specification, establishes a blank detail specification (BDS) for

- nanoporous activated carbon

used for

- electrochemical capacitors

Numeric values for the key control characteristics are left blank as they will be specified between customer and supplier in the detail specification (DS). In the DS key control characteristics can be added or removed if agreed between customer and supplier

2 Normative references

There are no normative references in this document.

3 Terms, definitions and abbreviated terms

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
nanomanufacturing
intentional synthesis, generation or control of nanomaterials, or fabrication step in the nanoscale, for commercial purposes

[SOURCE: ISO/TS 80004-1:2015, 2.11]

3.1.2
key control characteristic
KCC
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.

3.1.3
product specification
structured document which describes all characteristics of a product known to be relevant for applications of that product

3.1.4
blank detail specification
BDS
structured generic specification providing a comprehensive 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: Examples of nano-enabled products are: nanomaterials, nanocomposites and nano-subassemblies.

Note 2 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.

3.1.5
detail specification
DS
specification based on a blank detail specification with assigned values and attributes

Note 1 to entry: The properties listed in the detail specification are usually a subset of the key control characteristics listed in the relevant blank detail specification or sectional blank detail specification. The industrial partners define only those properties which are required for the intended application.

Note 2 to entry: Detail specifications are defined by the industrial partners. SDOs will be involved only if there is a general need for a detail specification in an industrial sector.

Note 3 to entry: The industrial partners may define additional key control characteristics if they are not listed in the blank detail specification or sectional blank detail specification.

3.1.6
measurand
quantity intended to be measured

Note 1 to entry: If the quantity is a key control characteristic, the measurement is an essential part of the quality management system.

3.1.7
measurement method
process of experimentally obtaining one or more values that can reasonably be attributed to a quantity

Note 1 to entry: If the quantity is a key control characteristic, the measurement is an essential part of the quality management system.

3.1.8
measurement principle
phenomenon serving as a basis of a measurement

EXAMPLE 1: Thermoelectric effect applied to the measurement of temperature.

EXAMPLE 2: Energy absorption applied to the measurement of amount-of-substance concentration.

EXAMPLE 3: Hall effect applied to the measurement of magnetic flux density.

Note 1 to entry: The phenomenon can be of a physical, chemical, or biological nature.

[SOURCE: IEC 60050-112:2010, 112-04-03]

3.1.9
measurement procedure
detailed description of a measurement according to one or more measurement principles and to a given measurement method, based on a measurement model, and including any calculation to obtain a measurement result

Note 1 to entry: A measurement procedure is usually documented in sufficient detail to enable an operator to perform a measurement.

Note 2 to entry: A measurement procedure can include a statement concerning a target measurement uncertainty.

Note 3 to entry: A measurement procedure is sometimes called a standard operating procedure, abbreviated SOP.

[SOURCE: ISO/IEC Guide 99:2007, 2.6]

3.1.10
measurement result
set of quantity values being attributed to a measurand together with any other available relevant information

Note 1 to entry: A measurement result is generally expressed as a single measured quantity value and a measurement uncertainty. If the measurement uncertainty is considered to be negligible for some purpose, the measurement result may be expressed as a single measured quantity value. In many fields, this is the common way of expressing a measurement result.

[SOURCE: ISO/IEC Guide 99:2007, 2.9, modified – Notes 1 and 3 to entry have been deleted.]

3.1.11
measurement accuracy
closeness of agreement between a measured quantity value and a true quantity value of a measurand

Note 1 to entry: The concept "measurement accuracy" is not a quantity and is not given a numerical quantity value. A measurement is said to be more accurate when it offers a smaller measurement error.

[SOURCE: ISO/IEC Guide 99, 2.13, modified – Note 2 and 3 to entry have been deleted.]

3.1.12
measurement standard
standardized measurement procedure
normative document established by consensus and approved by a recognized body, that provides a measurement procedure, for common and repeated use, aimed at the achievement of the optimum degree of order in a given context

Note 1 to entry: Standards are in general based on the consolidated results of science, technology and experience, and aimed at the promotion of optimum community benefits.

3.1.13
good practice guide
GPG
informal document which is not necessarily peer reviewed but can be used as a working document to establish a measurement procedure

Note 1 to entry: A GPG serves as the first document based on initial scientific research which is intended to be the first step toward future standardization.

3.1.14
standard maturity level
SML
measure for estimating the maturity of a measurement procedure based on the consensus achieved in the stakeholder community

Note 1 to entry: SML 1 – No documented measurement procedure available.

Note 2 to entry: SML 2 – Good practice guide publicly available based on a reasonable consensus achieved in the stakeholder community, e.g. an industrial or academic consortium.

Note 3 to entry: SML 3 – IEC or ISO standard or Technical Specification available which can be applied with modification and adaptation to the intended application and use case of the BDS scope.

Note 4 to entry: SML 4 – IEC or ISO standard or Technical Specification available for the exact intended application and use case of the BDS.

3.1.15
use case
specification of a generalized field of application, possibly entailing the following information about the system: one or several scenarios; the functional range; the desired behaviour; and the system boundaries

Note 1 to entry: The use case description typically does not include a detailed list of all relevant scenarios for this use case. Instead, a more abstract description of these scenarios is used.

[SOURCE: IEC TS 62565-1:2023, 3.18.]

3.1.16
procurement information
information other than key control characteristics needed for the procurement process.

3.2 Terms related to the nanoporous activated carbon

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
active material
material which electrostatically adsorbs and desorbs the ions at the electrode–electrolyte interface to store electric energy when the cell charges and discharges

3.2.3
nanopore
cavity with at least one dimension in the nanoscale, which may contain a gas or liquid

Note 1 to entry: The shape and content of the cavity can vary. The concept of nanopore overlaps with micropore (pore with width about 2 nm or less), mesopore (pore with width between approximately 2 nm and 50 nm), and macropore (pore with width greater than about 50 nm). See ISO 15901-3:2007.

Note 2 to entry: When nanopores are appropriately interconnected they may allow for transport through the material (permeability).

[SOURCE: ISO/TS 80004-4:2011, 2.13]

3.2.4
nanoporous material
solid material with nanopores

Note 1 to entry: The solid may be either amorphous, crystalline, or a mixture of both.

Note 2 to entry: The definitions of solid nanofoam (where most of the volume is occupied by pores) and nanoporous material (also materials with a small fraction of pores covered) are overlapping.

[SOURCE: ISO/TS 80004-4:2011, 3.4]

3.2.5
activated charcoal
activated carbon
carbon, usually in the form of granules, treated to enhance its surface area and consequent ability to adsorb and desorb the ions through a highly developed pore structure

3.2.6
nanoporous activated carbon
activated carbon with nanopores

Note 1 to entry: The performance of such activated carbon application mainly depends on its nanoporous structure.

3.3 Chemical key control characteristics

3.3.1
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 21268-2:2019, 3.6, modified – Note 1 to entry has been deleted.]

3.3.2
ash content
percent by mass of carbon-free residue on combustion and pyrolysis

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

3.3.3
metallic impurity
magnetic element, such as Fe, Co, Ni, present but not intentionally added to a material, and the minimum content of which is not controlled

3.3.4
elemental content
content of element, such as C, H, N, S, O, P, Si, within moisture free material, expressed in percent of mass

3.3.5
anion impurity
anion, such as Cl^-, SO_4^{2-}, NO_3^-, present but not intentionally added to a material, and the minimum content of which is not controlled

3.3.6
functional group
atom, or a group of atoms that has similar chemical properties whenever it occurs in different compounds, which defines the characteristic physical and chemical properties of families of organic compounds

[SOURCE: IEC TS 62607-6-13:2020, 3.1.2.2]

3.3.7
oxygen functional group
functional group containing oxygen atom

[SOURCE: IEC TS 62607-6-13:2020, 3.1.2.3]

3.4 Physical key control characteristic

3.4.1
particle size distribution
cumulative distribution of particle concentration as a function of particle size

[SOURCE: ISO 14644-1:2015, 3.2.4]

3.4.2
tap density
density of a powder in a container that has been tapped under specified conditions

[SOURCE: ISO 12749-3:2015, 3.4.4]

3.4.3
apparent density
loose bulk density
dry mass per unit volume of a powder obtained by free pouring under specified conditions

[SOURCE: ISO/TS 23362:2021, 3.1.4]

3.4.4
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 (Ω·m).

[SOURCE: ISO 14309:2019, 3.3]

3.4.5
pH value
measure of the concentration of acidity or alkalinity of a material in an aqueous solution

[SOURCE: ISO 20976-1:2019, 3.15]

3.4.6
circularity
degree to which the projected area of the particle is similar to a circle, based on its perimeter

3.5 Structural key control characteristics

3.5.1
specific surface area
absolute surface area of the sample divided by sample mass

[SOURCE: ISO 9277:2022, 3.11]

3.5.2
pore volume
volume of open pores unless otherwise stated

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

3.5.3
pore size distribution
percentage by numbers or by volume of each classified pore size which exists in a material

[SOURCE: ISO 3252:2019, 3.3.47]

3.5.4
crystal structure
arrangement of a regular and repeating internal unit of atoms in three dimensions in which the atoms are set in space in a fixed relation to each other

Note 1 to entry: For carbon material, interplanar spacing and crystallite size are important parameters to describe the crystal structure.

[SOURCE: ISO/TS 11937:2012, 3.4, modified – Note 1 to entry has been added.]

3.5.5
interplanar spacing
d_{hkl}
perpendicular distance between consecutive planes of the crystallographic plane set ($h\ k\ l$)

Note 1 to entry: For carbon material, especially amorphous carbon, interplanar spacing d_{002} of crystallographic plane set (002) is used to describe the distance between two adjacent graphene layers.

[SOURCE: ISO 25498:2018, 3.6, modified – Note 1 to entry has been added.]

3.5.6
crystallite size
dimensions of a single coherent crystalline region

Note 1 to entry: The mean size of crystal thickness L_c and crystal diameter L_a are often used to describe the crystallite size of carbon material.

3.6 Electrochemical key control characteristics

3.6.1
capacitance
ability of a capacitor to store electrical charge

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

[SOURCE: IEC 62576:2018, 3.5, modified – Information on units has been moved to a Note 1 to entry.]

3.6.2
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, modified – Information on units has been moved to a Note 1 to entry.]

3.6.3
specific capacitance
capacitance of capacitor divided by the mass or volume of active material mass

Note 1 to entry: Unit: farad per gram (F/g) or farad per cubic centimetre (F/cm^3).

3.6.4
leakage current
value of the current that flows through a capacitor after a charge for a fixed period of time

Note 1 to entry: Unit: ampere (A).

[SOURCE: IEC 62391-1:2022, 3.12, modified – Note 2 to entry has been deleted.]

3.6.5
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]

3.6.6
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

3.6.7
temperature endurance
ratio of the capacitance or internal resistance to its initial value after a specified charging time at constant voltage charge under a certain temperature

3.7 Measurement methods

3.7.1
constant current discharge
CCD
discharge during which the electric current is maintained at a constant value regardless of the battery voltage or temperature

3.7.2
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]

3.7.3
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]

3.8 Symbols and abbreviated terms

ICP	inductively coupled plasma
MS	mass spectrometry
OES	optical emission spectroscopy
XRF	X-ray fluorescence
AAS	atomic absorption spectrometry
XPS	X-ray photoelectron spectroscopy
FT-IR	Fourier transform infrared spectroscopy
SEM	scanning electron microscopy
XRD	X-ray diffraction
GCD	galvanostatic charge and discharge
GC	galvanostatic charge
CV	constant voltage
S	surface area
C	circularity
X_{Fmax}	maximum Feret diameter
EDLC	electric double layer capacitor
IGD	infrared gas detector
TCD	thermal conductivity detector

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 the nanoporous activated carbon 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, the users of the detail specification shall agree bilaterally on it. For the KCCs in this document, Clause 6 provides a general overview and Annex A provides supporting information for standardized KCC measurement procedures.

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 or 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 and 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 Recommended nanoporous activated carbon specification format

5.1 General product description and procurement information

General product description and procurement information about the nanoporous activated carbon for electrochemical capacitors should be provided by the manufacturer or product supplier according to Table 1.

Table 1 – Format for general procurement information

Item No.	Item		Information	
1.1	Supplier			
1.2	Product trade name			
1.3	ID number			
1.4	Manufacturing method			
1.5	Description of the manufacturing process available	☐ No		
		☐ Yes	Process reference	
1.6	Technical drawing available	☐ No		
		☐ Yes	Drawing number	
1.7	Typical batch quantity	☐ Weight [kg]		
1.8	Traceability requirements	☐ Batch number ☐ Serial number ☐ Others, specify		
		Manufacturing date		
1.9	Specification	Number		
		Revision level		
		Date of issue		
1.10	Packaging requirements or handling instructions	☐ No		
		☐ Yes	Instruction number	
1.11	Material Safety Data Sheet (MSDS) available	☐ No		
		☐ Yes	MDS number	
1.12	Factory name and location			

5.2 Chemical key control characteristics

Chemical characteristics as detailed in Table 2 shall be agreed between manufacturer and user.

Table 2 – Format for chemical key control characteristics

KCC No.	KCC	SPECIFICATION	UNIT	MEASUREMENT METHOD	SML	MEASUREMENT PROCEDURE
2.1	Water content	Less than [ ]	% mass fraction	Karl Fischer	4	IEC TS 62607-4-8 [5] (Clause A.2)
				Drying loss	4	ISO 21340 [6] (Clause A.3)
2.2	Ash content	Less than [ ]	% mass fraction	Incineration	4	ISO 21340 [6] (Clause A.4)
2.3	Metallic impurities	Fe: Less than [ ] Co: Less than [ ] Ni: Less than [ ] K: Less than [ ] Na: Less than [ ] Mg: Less than [ ] Ca: Less than [ ]	ppm	ICP-MS	3	IEC TS 62607-6-20 [7] (Clause A.5)
				ICP-OES	1	ISO 19050 [8] (Clause A.6)
2.4	Anion impurities	F^-: Less than [ ] Cl^-: Less than [ ] Br^-: Less than [ ] SO_4^{2-}: Less than [ ] NO_3^-: Less than [ ]	ppm	Ion chromatography	1	Not available (Clause A.7)
2.5	Elemental content	C: [ ] ± [ ] H: [ ] ± [ ] N: [ ] ± [ ] S: [ ] ± [ ] O: Less than [ ]	% mass fraction	Elemental analyser	3	ISO 21663 [9] (Clause A.8)
				CS analyser, ONH analyser	3	IEC TS 62607-6-19 [10] (Clause A.9)
2.6	Oxygen functional groups	Carboxyl: Less than [ ] Lactone: Less than [ ] Phenol: Less than [ ] Carbonyl: Less than [ ]	mmol/g	Boehm titration	3	IEC TS 62607-6-13 [11] (Clause A.10)
				XPS	3	ISO 16243 [12] (Clause A.11)

5.3 Physical key control characteristics

Physical characteristics as detailed in Table 3 shall be agreed between manufacturer and user.

Table 3 – Format for physical key control characteristics

KCC No.	KCC	SPECIFICATION	UNIT	MEASUREMENT METHOD	SML	MEASUREMENT PROCEDURE
3.1	Particle size distribution	$D10$: [ ] ± [ ] $D50$: [ ] ± [ ] $D90$: [ ] ± [ ]	µm	Laser diffraction methods	4	ISO 13320 [13] (Clause A.12)
3.2	Tap density	Greater than [ ]	g/cm^3	Tapping method	4	ISO 3953 [14] (Clause A.13)
3.3	pH value	[ ] ± [ ]		pH meter	4	ISO 21340 [6] (Clause A.14)
3.4	Circularity	[ ] ± [ ]		Static image analysis method	3	ISO 13322-1 [15] (Clause A.15)
				Dynamic image analysis method	3	ISO 13322-2 [16] (Clause A.16)
				SEM	3	ISO 19749 [17] (Clause A.17)
3.5	Apparent density	[ ] ± [ ]	g/cm^3	Funnel method	4	ISO 3923-1 [18] (Clause A.18)
3.6	Volume resistivity	[ ] ± [ ]	Ω·cm	Four probe method	4	IEC TS 62607-6-1 [19] (Clause A.19)

5.4 Structural key control characteristics

Structural characteristics as detailed in Table 4 shall be agreed between manufacturer and user.

Table 4 – Format for structural key control characteristics

KCC No.	KCC	SPECIFICATION	UNIT	MEASUREMENT METHOD	SML	MEASUREMENT PROCEDURE
4.1	Specific surface area	[ ] ± [ ]	m^2/g	Gas adsorption	3	ISO 21340 [6] (Clause A.20)
4.2	Pore volume	Greater than [ ]	cm^3/g	Gas adsorption	4	ISO 21340 [6] (Clause A.21)
4.3	Pore size distribution	Pore size distribution curve		Gas adsorption	4	ISO 15901-2 [20] (Clause A.22)
4.4	Crystal structure	d_{002}: [ ] ± [ ] L_a: [ ] ± [ ] L_c: [ ] ± [ ]	nm	XRD	1	Not available (Clause A.23)
4.5	Defect level	I_D/I_G: [ ] ± [ ]		Raman spectra	1	Not available (Clause A.24)

5.5 Electrochemical key control characteristics

Electrochemical characteristics of nanoporous activated carbon as detailed in Table 5 shall be agreed between manufacturer and user.

Table 5 – Format for electrochemical key control characteristics

KCC No.	KCC	SPECIFICATION	UNIT	MEASUREMENT METHOD	SML	MEASUREMENT PROCEDURE
5.1	Specific capacitance	Greater than [ ]	F/g	Constant current charge (CCC), constant voltage charge(CVC) and constant current discharge (CCD) using coin or three-electrode or cylindrical type EDLC	1	Not available (Clause A.25)
5.2	Internal resistance	Less than [ ]	Ω	CCC-CVC-CCD using coin or three-electrode or cylindrical type EDLC	1	Not available (Clause A.26)
5.3	Voltage maintenance rate	Greater than [ ]	%	CCC-CVC using coin or three-electrode or cylindrical type EDLC	1	Not available (Clause A.27)
5.4	Leakage current	Less than [ ]	mA	CCC-CVC using coin or three-electrode or cylindrical type EDLC	1	Not available (Clause A.28)
5.5	Endurance in cycling	Greater than [ ] after [ ] cycles	%	CCC-CCD using coin or three-electrode or cylindrical type EDLC	1	Not available (Clause A.29)
5.6	Temperature endurance	Greater than [ ] after [ ] hours at [ ] °C	%	CVC using coin or three-electrode or cylindrical type EDLC	1	Not available (Clause A.30)

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 procedures which are summarized in the overview Table 6 for each combination of KCC and measurement method.

SML 1: A standardized measurement procedure 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.

SML 2: A standardized measurement procedure 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 GPD is publicly available, it can be referenced instead.

SML 3: A standardized measurement procedure 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.

SML 4: A standardized measurement procedure 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 the column "measurement procedure" of the KCC tables.

NOTE In the cases of SML 2 and SML 3 it is suggested to transform the measurement protocol into a documented standard and to perform all steps to prepare issuing a New Work Item Proposal to the IEC through the appropriate National Committee.

Table 6 – Matrix of properties and methodologies of nanoporous activated carbon for electrochemical capacitors

Chemical KCC	Key control characteristic (KCC)							
Water content	Karl Fischer method	4						IEC TS 62607-4-8
Ash content	Incineration	4	4					ISO 21340
Magnetic impurities	ICP-MS			3				IEC TS 62607-6-20
	ICP-OES			3				ISO 19050
Anion impurities	Ion chromatography				1			Not available
Elemental content	Elemental analyser					3		ISO 21663
	CS/ONH analyser					3		IEC TS 62607-6-19
Oxygen functional groups	Boehm titration						3	IEC TS 62607-6-13
	XPS						3	ISO 16243
	Laser diffraction							ISO 13320
	Tapping method							ISO 3953
	pH meter							ISO 21340
	Static image analysis methods							ISO 13322-1
	Dynamic image analysis methods							ISO 13322-2
	SEM							ISO 19749
	Funnel method							ISO 3923-1
	Four probe method							IEC TS 62607-6-1
	Gas adsorption							ISO 15901-2
	XRD							Not available
	Raman spectra							Not available
	CCC-CVC-CCD							Not available
	CCC-CVC-CCD							Not available
	CCC-CVC							Not available
	CCC-CVC							Not available
	CCC-CCD							Not available
	CVC							Not available

Key control characteristic (KCC)		Physical KCC						Structural KCC				
		Particle size distribution	Tap density	pH value	Circularity	Apparent density	Volume resistivity	Specific surface area	Pore volume	Pore size distribution	Crystal structure	Defect level
Karl Fischer method	IEC TS 62607-4-8											
Incineration	ISO 21340							3	4			
ICP-MS	IEC TS 62607-6-20											
ICP-OES	ISO 19050											
Ion chromatography	Not available											
Elemental analyser	ISO 21663											
CS/ONH analyser	IEC TS 62607-6-19											
Boehm titration	IEC TS 62607-6-13											
XPS	ISO 16243											
Laser diffraction	ISO 13320	4										
Tapping method	ISO 3953		4									
pH meter	ISO 21340			4								
Static image analysis methods	ISO 13322-1				3							
Dynamic image analysis methods	ISO 13322-2				3							
SEM	ISO 19749				3							
Funnel method	ISO 3923-1					4						
Four probe method	IEC TS 62607-6-1						4					
Gas adsorption	ISO 15901-2									4		
XRD	Not available										1	
Raman spectra	Not available											1
CCC-CVC-CCD	Not available											
CCC-CVC-CCD	Not available											
CCC-CVC	Not available											
CCC-CVC	Not available											
CCC-CCD	Not available											
CVC	Not available											

Key control characteristic (KCC)		Electro-chemical KCC						
		Specific capacitance	Internal resistance	Voltage maintenance rate	Leakage current	Endurance in cycling	Temperature Endurance
IEC TS 62607-4-8	Karl Fischer method						
ISO 21340	Incineration						
IEC TS 62607-6-20	ICP-MS						
ISO 19050	ICP-OES						
Not available	Ion chromatography						
ISO 21663	Elemental analyser						
IEC TS 62607-6-19	CS/ONH analyser						
IEC TS 62607-6-13	Boehm titration						
ISO 16243	XPS						
ISO 13320	Laser diffraction						
ISO 3953	Tapping method						
ISO 21340	pH meter						
ISO 13322-1	Static image analysis methods						
ISO 13322-2	Dynamic image analysis methods						
ISO 19749	SEM						
ISO 3923-1	Funnel method						
IEC TS 62607-6-1	Four probe method						
ISO 15901-2	Gas adsorption						
Not available	XRD						
Not available	Raman spectra						
Not available	CCC-CVC-CCD	1					
Not available	CCC-CVC-CCD		1				
Not available	CCC-CVC			1			
Not available	CCC-CVC				1		
Not available	CCC-CCD					1	
Not available	CVC						1

Annex A
(normative)

Supporting information for standardized KCC measurement procedures

A.1 General

Annex A provides supporting information for standardized KCC measurement procedures in the following cases.

SML 3: A standardized measurement procedure 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 the column "measurement procedure" of the KCC tables.

SML 4: A standardized measurement procedure 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 the column "measurement procedure" of the KCC tables.

A.2 Water content: Karl Fischer

A.2.1 General

The water content of a nanoporous activated carbon can be determined by either Karl Fischer method or drying loss method. Drying loss method is preferred when the water content is larger than 1 % mass fraction, while Karl Fischer method is preferred when the water content is less than 0,1 % mass fraction.

In the Karl Fischer method, water (H_2O) reacts quantitatively with iodine (I_2) and sulfur dioxide (SO_2) in the presence of a lower alcohol such as methanol (CH_3OH)) and an organic base (RN) to produce iodide ion. In coulometric Karl Fischer titration, iodine (I_2) is generated electrochemically from iodide (I^-). The amount of water in the sample is calculated by measuring the current needed for the electrochemical generation of iodine (I_2) from iodide (I^-).

A.2.2 Measurement standard

IEC TS 62607-4-8 [5] is directly applicable to the nanoporous activated carbon.

A.3 Water content: Drying loss

A.3.1 General

The water content of a nanoporous activated carbon can be determined by either Karl Fischer method or drying loss method. Drying loss method is preferred when the water content is larger than mass fraction 1 %, while Karl Fisher method is preferred when the water content is less than mass fraction 0,1 %.

For drying loss method, dry a certain mass of sample in a 110 °C to 120 °C thermostatic dryer for approximately 3 h or longer, then weigh the mass after cooling the sample in a desiccator. Finally the water content in a sample can be calculated from the loss of mass on drying.

A.3.2 Measurement standard

ISO 21340 [6] is directly applicable to the nanoporous activated carbon.

A.4 Ash content: Incineration

A.4.1 General

The ash content can quickly reflect the total purity of a nanoporous activated carbon. One gram of sample is heated at 800 °C to 900 °C for 1 h in an oxygen atmosphere, the organic elements, such as C, N, H, O and S, will be combusted to produce gas CO_2, NO_2, H_2O and SO_2, respectively. The inorganic elements, such as Fe, Co, Ni, Cu and Zn, will be combusted to produced oxide or salt, which will deposit in the crucible to form residue ash. The ash content is derived by residue obtained after incineration, being divided by the mass of the dried test portion.

A.4.2 Measurement standard

ISO 21340 [6] is directly applicable to the nanoporous activated carbon.

A.5 Metallic impurities: ICP-MS

A.5.1 General

Generally, the metallic impurities in an aqueous solution can be determined by ICP-MS. Therefore, a nanoporous activated carbon powder shall be pre-treated to be a solution, then the metallic element contents in the sample solution shall be determined using an ICP-MS instrument.

A.5.2 Measurement standard

IEC TS 62607-6-20 [7] was originally developed for graphene-based materials; it can be used with care also for other materials covered by this document.

A.5.3 Adaptations and modifications required

Adaptation: At least 50 mg of sample nanoporous activated carbon are needed to digest in 5 mL of HNO_3.

A.6 Metallic impurities: ICP-OES

A.6.1 General

Generally, the metallic impurities in an aqueous solution can be determined by ICP-OES. Therefore, a nanoporous activated carbon powder shall be pre-treated to be a solution, then the metallic element contents in the sample solution shall be determined by the ICP-OES.

A.6.2 Measurement standard

ISO 19050 [8] was originally developed for rubber, raw, vulcanized; it can be used with care also for other materials covered by this document.

A.6.3 Adaptations and modifications required

Adaptation: Three commonly used methods of destruction of organic matter are involved in ISO 19050, including wet oxidation, drying ashing and microwave digestion. For a nanoporous activated carbon, microwave digestion is preferred.

A.7 Anion impurities: Ion chromatography

A.7.1 General

The anions in a nanoporous activated carbon are extracted by boiling or ultrasonicating the mixture of 1 g of sample and 40 ml to 50 ml water for several minutes, then the anion content in the filtrate can be determined by ion chromatography.

A.7.2 Measurement standard

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

A.8 Elemental content: Elemental analyser

A.8.1 General

The elemental content of a nanoporous activated carbon refers to the content of C, H, N, S and O. The elemental content can be determined by an elemental analyser. A known mass of sample is held in a tin container and then dropped inside the quartz tube furnace at no less than 1 000 °C in an oxygen stream for complete oxidation to convert into gaseous products of combustion. These gaseous products consist mainly of carbon dioxide, water vapour, elemental nitrogen and/or nitrogen oxides, oxyacids and oxides of sulfur and hydrogen halides. The products are converted to water vapour, nitrogen, carbon dioxide and sulfur dioxide. The fractions of carbon dioxide, water vapour, nitrogen and sulfur dioxide in the gas stream are separated by trapping or suitable chromatographic column, then determined quantitatively by appropriate instrumental gas analysis procedures. Thus, the corresponding C, H, N and S content can be calculated.

A.8.2 Measurement standard

ISO 21663 [9] was originally developed for solid recovered fuels; it can be used with care also for other materials covered by this document.

A.8.3 Adaptations and modifications required

Adaptation: The minimum laboratory sample amount for the analysis of solid recovered fuels (SRF) is 100 g. Compared with the SRF, the nanoporous activated carbon is more uniform, so the minimum laboratory sample amount for the nanoporous activated carbon can be less than 1 g.

A.9 Elemental content: CS analyser, ONH analyser

A.9.1 General

The elemental content can be determined by CS analyser and ONH analyser. For the CS analyser, the test sample is combusted by heating in a resistance furnace in a pure oxygen atmosphere, causing sulfur to react to sulfur dioxide (SO_2) and carbon to carbon monoxide (CO) and carbon dioxide (CO_2). The carbon content or sulfur content is detected by IGD using infrared absorption method.

The ONH analyser is based on the impulse heating inert gas fusion principle, which involves fusion of the test sample in a single-use graphite crucible under helium (He) or argon (Ar) gas at a high temperature (e.g. 2 400 °C). The oxygen content of the sample reacts with carbon from the graphite crucible to CO or little CO_2, the nitrogen content to elemental N_2, and the hydrogen content to H_2. The CO and CO_2 concentration is analysed by IGD and the O content is calculated. The N_2 concentration is measured by a TCD. Then the N content can be derived from a detectable signal generated by the TCD. For the hydrogen content measurement by a TCD, the carrier gas shall be nitrogen or Ar gas because of the large difference in thermal conductivity between N_2 (or Ar) and H_2, and copper oxide shall be replaced with Schutze's reagent, which is made up of iodine pentoxide, I_2O_5, and sulfuric acid on granular silica gel. Schutze's reagent can convert CO to CO_2 at room temperature without converting H_2 to H_2O. The hydrogen content can also be obtained by an IGD, which measures the content of converted H_2O.

A.9.2 Measurement standard

IEC TS 62607-6-19 [10] was originally developed for solid recovered fuels; it can be used with care also for other materials covered by this document.

A.9.3 Adaptations and modifications required

Adaptation: Due to the low bulk density of graphene powder, which is several grams per litre, a mass of at least 20 mg shall be pressed with pelletizer under 3 MPa to 5 MPa to avoid sample splatter during combustion. However, the bulk density of the nanoporous activated carbon is about 0,4 g/cm^3, so it can be directly tested without any press.

A.10 Oxygen functional groups: Boehm titration

A.10.1 General

Boehm titration is a quantitative analysis method to determine the oxygen functional groups on carbon material. Oxygen functional groups on the carbon material of different acidities can be neutralized by bases of different strengths. Sodium ethoxide is the strongest base used in this method that can neutralize acids including carboxyl groups, lactone groups, hydroxyl groups and reactive carbonyl groups. Sodium hydroxide is the second strongest base that can neutralize carboxyl groups, lactone groups and hydroxyl groups. Sodium carbonate neutralizes lactone groups and carboxyl groups. And sodium bicarbonate neutralizes carboxyl groups only. Therefore, the content of each type of oxygen functional group can be determined from the difference between the normalized base consumptions, which are derived by dividing total base consumption by mass of reacting sample.

A.10.2 Measurement standard

IEC TS 62607-6-13 [11] was originally developed for graphene powder; it can be used with care also for other materials covered by this document.

A.10.3 Adaptations and modifications required

1) Adaptation 1: In IEC TS 62607-6-13, 0,15 g to 0,5 g of graphene powder sample react with base solution. For the nanoporous activated carbon, 1 g to 2 g of sample are needed to react with base solution.
2) Adaptation 2: In IEC TS 62607-6-13, the mixture of sample and base solution are shaken for at least 3 h at (25 ± 2) °C using an oscillator. For the nanoporous activated carbon, the mixture of sample and base solution are shaken for at least 24 h at (25 ± 2) °C using an oscillator.

3) Adaptation 3: In IEC TS 62607-6-13, the water solutions are suction-filtrated and the ethanol solutions are centrifuged to separate the sample from the base solution after the shaking. For the nanoporous activated carbon, all solutions are centrifuged to separate the sample from the base solution.

A.11 Oxygen functional groups: XPS

A.11.1 General

XPS is a qualitative and semi-quantitative method to determine the oxygen functional groups on several nanometre depth surface of a material. Oxygen functional groups of a nanoporous activated carbon can be obtained through fitting the survey and a high-resolution spectrum of O 1s and C 1s.

A.11.2 Measurement standard

ISO 16243 [12] can be used with care.

A.11.3 Adaptations and modifications required

Adaptation: ISO 21340 [6] is a general method to record and report data in XPS. For a specific nanoporous activated carbon, the binding energy of different kinds of oxygen functional groups and their fitting process shall be optimized.

A.12 Particle size distribution: Laser diffraction method

A.12.1 General

Laser diffraction is the common method to determine the particle size distribution. According to this method, the angular distribution of the intensity of scattered light by a particle (scattering pattern) is dependent on the particle size. When the scattering is from a cloud or ensemble of particles the intensity of scattering for any given size class is related to the number of particles and their optical properties, present in that size class. A nanoporous activated carbon, dispersed at an adequate concentration in a suitable liquid or gas, is passed through the beam of a monochromatic light source, usually a laser. The light scattered by the particles is measured by an array of photo detectors. The numerical values from each detector are recorded for subsequent analysis. The theoretical scattering patterns of unit volumes of particles in selected size classes are used to build a matrix and together with a mathematical procedure are used to solve the inverse problem, providing a volumetric particle size distribution (PSD), iterated to provide a best fit to the measured scattering pattern.

A.12.2 Measurement standard

ISO 13320 [13] is directly applicable to the nanoporous activated carbon.

A.13 Tap density: Tapping method

A.13.1 General

The tap density of the nanoporous activated carbon will affect the power density of the electrochemical capacitor. When determining the tap density of a nanoporous activated carbon, a specified amount of sample powder in a 25 ml or 100 ml is tapped by means of tapping apparatus until no further decrease in the volume of the powder takes place. The mass of the powder divided by its volume after the test gives its tap density.

A.13.2 Measurement standard

ISO 3953 [14] is directly applicable to the nanoporous activated carbon.

A.14 pH value: pH meter

A.14.1 General

Place 0,5 g of dry sample in a 200 ml beaker, add 100 ml of water, heat gently and boil for 5 min. After cooling to room temperature, filter the sample. Add water to the filtrate to give a volume of 100 ml, and measure the pH value of filtrate using a pH meter.

A.14.2 Measurement standard

ISO 21340 [6] is directly applicable to the nanoporous activated carbon.

A.15 Circularity: Static image analysis method

A.15.1 General

A certain amount of a nanoporous activated carbon particles is appropriately dispersed and fixed in the object plane of the analysis instrument. Then images of these particles are acquired by the imaging instrument. The particle shape, including the circularity and the Feret diameter, are analysed based on these images.

A.15.2 Measurement standard

ISO 13322-1 [15] can be used with care also for other materials covered by this document.

A.15.3 Adaptations and modifications required

Adaptation: ISO 13322-1 is to give guidance when using images for particle size analysis. For the nanoporous activated carbon, the detail parameter for the test shall be optimized.

A.16 Circularity: Dynamic image analysis method

A.16.1 General

Dynamic image analysis method entails using techniques for dispersing particles in liquid or gas, taking in-focus, still images of them while the particles are moving and subsequently analysing the images.

A.16.2 Measurement standard

ISO 13322-2 [16] can be used with care also for other materials covered by this document.

A.16.3 Adaptations and modifications required

Adaptation: ISO 13322-2 is to provide guidance for measuring and describing particle size distribution, using image analysis methods where particles are in motion. For a nanoporous activated carbon, the detail parameter for the test shall be optimized.

A.17 Circularity: SEM

A.17.1 General

The images of a nanoporous activated carbon sample particles are acquired by the SEM. The particle shape analysis is based on these SEM images. The particles shall not be overlapped with each other.