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TECHNICAL SPECIFICATION

colour inside

Nanomanufacturing – Key control characteristics – Part 4-6: Nano-enabled electrical energy storage – Determination of carbon content for nano-enabled electrode materials, infrared absorption method

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

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International Standard IEC 62607-4-6 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
113/379/DTS	113/402/RVDTS

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 document 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 document will remain unchanged until the stability date indicated on the IEC website under "http://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- withdrawn,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.

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

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INTRODUCTION

Electrical energy storage devices have been utilized in many applications such as portable electronics, electric vehicles and aircraft. Rapid development in these markets poses great demand for high-performance devices, in which the main properties are determined by their electrode materials.

Carbon content has significant influence on the performance and quality of electrode material. Carbon serves as a conductive agent improving interparticle electron conduction performance, therefore a suitable amount of carbon is necessary. When its amount is not enough, it is possible the conductivity will not improve effectively and this causes high internal resistance, low discharge platform or low capacity, which can result in bad rate performance and cycling life. On the other hand, high carbon content can have a tendency to aggregate and be hard to separate, which may introduce factory processing problems. Superfluous carbon can influence the power density of the batteries as carbon has relatively low specific capacity.[1][2]1

However, nano-sized powder can be easily flowed away with gas flow in the furnace chamber, which influences the determination of carbon content and may contaminate the test system.

This document provides a method to measure the carbon content of nano electrode materials that will be employed in electrical energy storage devices, and to evaluate the best combinations of composite material recipes of nano electrodes. Following this method will allow comparison of the results of different research groups.

This method is intended for comparing the carbon content of composite materials with cathode nanomaterials in the study stage, not for evaluating the electrode in end products.

The method is applicable for nano materials exhibiting function or performance only possible with nanotechnology, intentionally added to composite materials for improvement in the performance of electrical energy storage devices.

1 Numbers in square brackets refer to the Bibliography.

NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 4-6: Nano-enabled electrical energy storage – Determination of carbon content for nano-enabled electrode materials, infrared absorption method

1 Scope

This part of IEC 62607, which is a Technical Specification, provides a method for determination of carbon content of nano electrode materials by infrared absorption spectroscopy method. The method is applicable to carbon contents of mass fraction between 0,001 % and 100 %.

This method will enable customers to:

a) decide whether or not a nano electrode material is usable, and

b) select a nano electrode material with suitable carbon content for its application.

This document includes:

– recommendations for sample preparation,

– outlines of the experimental procedures used to measure electrode nanomaterial properties,

– methods of interpretation of results and discussion of data analysis, and

– case studies.

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 62607-4-2:2016, *Nanomanufacturing – Key control characteristics – Part 4-2: Nano-enabled electrical energy storage – Physical characterization of cathode nanomaterials, density measurement*

ISO/TS 80004-1:2010, *Nanotechnologies – Vocabulary – Part 1: Core terms*

3 Terms, definitions and abbreviated terms

3.1 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO/TS 80004-1 and the following apply.

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

3.1.1
electrode nanomaterial
material used in nano-enabled energy storage devices such as lithium-ion batteries or super capacitors, which contains a fraction of nanomaterial and exhibits function or performance made possible only with the application of nanotechnology [3].

[SOURCE: IEC TS 62607-4-3:2015, 3.1.1]

Note 1 to entry: In this document, it refers to the raw material powders (e.g. LCO, NCA, NCM, and LFP) without any additives (e.g. carbon nanomaterials like CB, carbon nanotubes or fibres) or organic binder (e.g. PVDF or SBR).

3.2 Abbreviated terms

IR	infrared
LCO	lithium cobalt oxide, $LiCoO_2$
NCA	lithium nickel cobalt aluminium oxide, $Li(Ni_xCo_yAl_{1-x-y})O_2$
NCM	lithium nickel cobalt manganese oxide, $Li(Ni_xCo_yMn_{1-x-y})O_2$
LFP	lithium iron phosphate, $LiFePO_4$
CB	carbon black
PVDF	polyvinylidene difluoride
SBR	styrene-butadiene rubber

4 Reagents and materials

4.1 Analysis gas

Oxygen (O_2), of commercial grade, higher than 99,5 % purity in volume (V_i/V_{tot}).

NOTE The analysis gas is utilized to oxidize carbon of the sample.

4.2 Carrier gas

Oxygen (O_2) or inert gas (N_2、Ar), of commercial grade, higher than 99,5 % purity in volume (V_i/V_{tot}).

NOTE Carrier gas is utilized to carry the carbon dioxide into the analytical host. It could be the same as the analysis gas or be different, according to the instrument model.

4.3 Fluxes

Tungsten particles (W), tin particles (Sn) and pure iron (Fe), of known low carbon content less than 0,000 8% in mass (m_i/m_{tot}). The particle size should be approximately 420 µm to 840 µm (20 mesh to 40 mesh).

NOTE Tungsten particles are necessary while the other two, which work as accelerators, are not.

4.4 Certified reference materials

Steel and iron certified reference materials or coal certified reference materials. The certified reference materials should have similar or higher carbon content than the sample.

5 Apparatus

5.1 Analytical balance

The analytical balance used should have a resolution of 0,001 g.

5.2 Powder compressor

The powder compressor should consist of two parts: (1) die, and (2) die compressor.

5.3 High-frequency infrared ray carbon/sulfur analyzer

The analyzer typically consists of three parts, which are analytical host, analytical balance and computer. The analytical host consists of high-frequency induction furnace, gas absorption cell and gas supply system. The recommended working conditions are given in Table 1.

Table 1 – Recommended working conditions of high frequency infrared carbon and sulfur analyzer

Instrument operating conditions	Parameter values
Analysis gas: Oxygen (O_2)	99,5 %
Input oxygen pressure	0,35 MPa to 0,40 MPa
Carrier gas: Oxygen (O_2) or insert gas (Ar/N_2)	99,5 %
Input carrier gas pressure	0,25 MPa to 0,30 MPa
Analysis gas flow	3,0 l/min to 4,0 l/min
Carrier gas flow	1,0 l/min to 2,0 l/min
High frequency induction furnace power	5 kVA
Analysis time	30 s to 60 s

5.4 Muffle furnace

The muffle furnace should be capable of maintaining a temperature of (1 200 ± 50) ºC.

5.5 Crucibles

The crucibles should be ceramic- (for example alumina and zirconia), platinum- or quartz-encapsulated tungsten with carbon content lower than 0,002 %. Before each test, the crucibles shall first be calcined in a suitable muffle furnace for 8 h at (1 000 ± 50) ºC, then removed from the furnace and placed in a desiccator to cool down in a moisture-free atmosphere.

6 Test methods

6.1 General

The tableted sample is mixed with fluxing agent, then it is combusted to a temperature above 1 000 ºC in the high-frequency induction furnace, under pure oxygen atmosphere, to convert any carbon compounds to carbon dioxide gas. The resulting carbon dioxide gas is filtered and dried before IR measurement. Infrared spectroscopy is used to measure the carbon dioxide signal at 4,26 µm (see the gas path diagram and the light path diagram in Annex A). The result is converted into carbon content (%) of the material.

6.2 Sample preparation

6.2.1 Drying

Dry the sample at 105 ºC for two hours (for flammable samples, special care shall be paid and corresponding requirements followed, to prevent damage). Put it into a desiccator and cool it down to room temperature.

6.2.2 Tableting

Weigh a certain mass of sample, use a powder compressor to compress the dried sample, ensure the sample be compressed to a tablet and the tablet will not fall apart by holding with tweezers; IEC TS 62607-4-2:2016, 5.1.3 applies.

6.3 Start up

The apparatus shall be started up and checked, and stabilized before measurements according to the instructions of the apparatus.

6.4 Blank verification and determination

Prior to determination, carry out a test without any sample in the crucible.

a) Using the measurement spoon, put an appropriate amount of tungsten flux particles according to the instructions of the apparatus (suggested range 1,5 g to 2,0 g) into a calcined crucible. Place the crucible in the induction furnace and perform the measurement.

b) Perform three measurements as described in 6.4 a).

c) Based on the three determinations, adjust the analyzer to zero setting and calculate the acceptance criteria for blank verification.

The value obtained shall correspond to the acceptance criteria specified for the blank determination; if not, readjust the blank setting.

6.5 Checking and calibration

a) Weigh 0,1 g of the certified reference materials, and transfer to a calcined crucible. Add an appropriate amount of tungsten flux particles according to the instructions of the apparatus (suggested range 1,5 g to 2,0 g) into the crucible. Place the crucible in the furnace after the samples are well blended and perform the measurement.

b) Perform three measurements as described in 6.5 a). The value obtained shall correspond to the acceptance criteria specified for calibration check; if not, recalibrate the system.

c) Based on the three determinations, calibrate the analyzer.

6.6 Analysis

a) Weigh about 0,1 g of the tableted sample, and transfer to the calcined crucible. Add an appropriate amount of tungsten flux particles according to the instructions of the apparatus (suggest range 1,5 g to 2,0 g) into the crucible.

b) Place the crucible in the furnace after the samples are well blended and perform the measurement.

c) Record the test result and repeat the measurement two more times.

6.7 Reporting the results

The final result is the expression of the mean value of the measurements performed on the sample. The carbon content shall be expressed as a percentage (%).

7 Data verification

The difference between two independent results, found on identical test materials, conducted by one analyst using the same apparatus within a short time interval should not exceed 5 % of the carbon content.

8 Test report

The test report shall include the following information:

a) the method used and the reference to this document;

b) the results and the form in which they are expressed;

c) any unusual features noted during the determination;

d) any operation not specified in this document that may influence the results.

Annex A
(informative)

Case study

A.1 The gas path diagram and the light path diagram

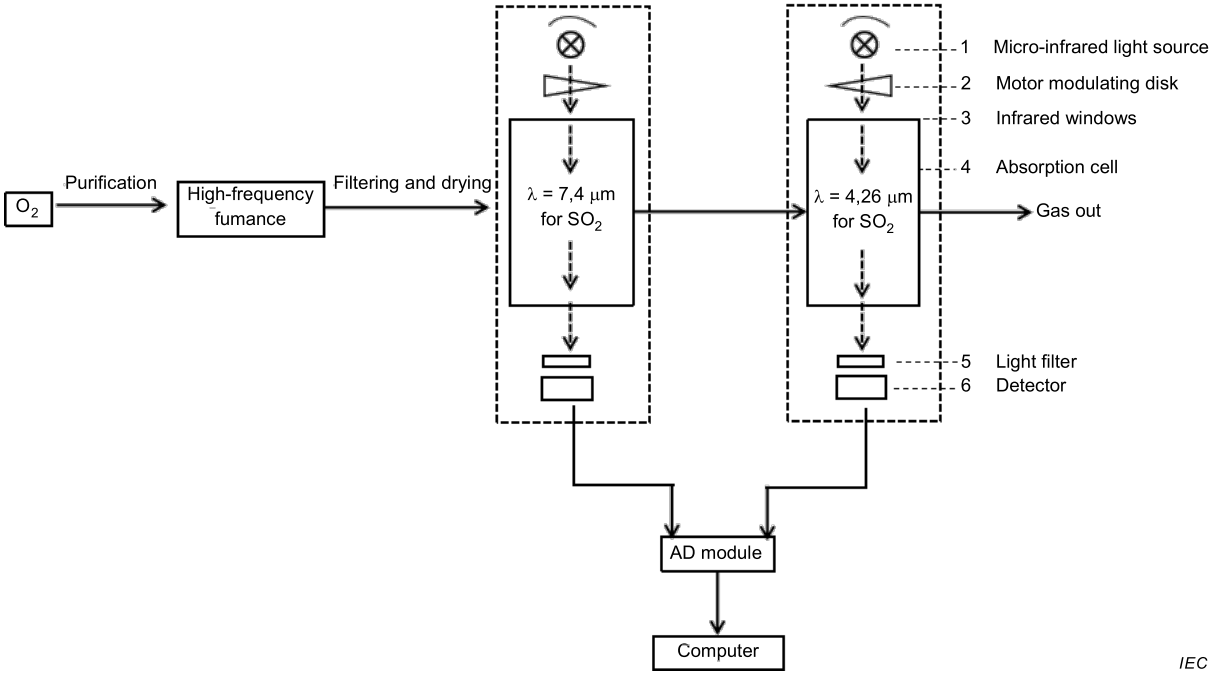


Figure A.1 – The gas and light (dashed box) path diagram

The carbon element in the sample is burned to CO_2 with abundant O_2 gas. The resultant gas mixture is then transferred to the IR absorption cell, and the infrared absorption at 4,26 μm of the CO_2 is measured (see Figure A.1). The absorbance of CO_2 follows Beer's law.

$$I = I_0 \exp(-aPL)$$

where

I_0 is the incident intensity;

I is the transmitted intensity;

a is the absorption coefficient with the unit of $m^{-1} \cdot Pa^{-1}$;

P is the partial pressure of the CO_2 gas with the unit of Pa;

L is the length of the absorption cell with the unit of m.

A.2 Procedures of sample preparation and carbon content determination

Procedures of sample preparation and carbon content determination are shown in Figure A.2.

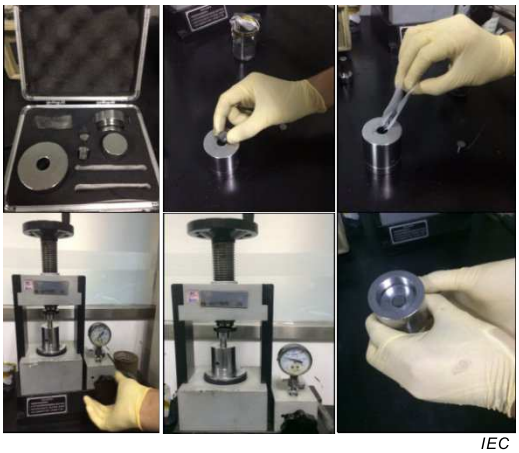
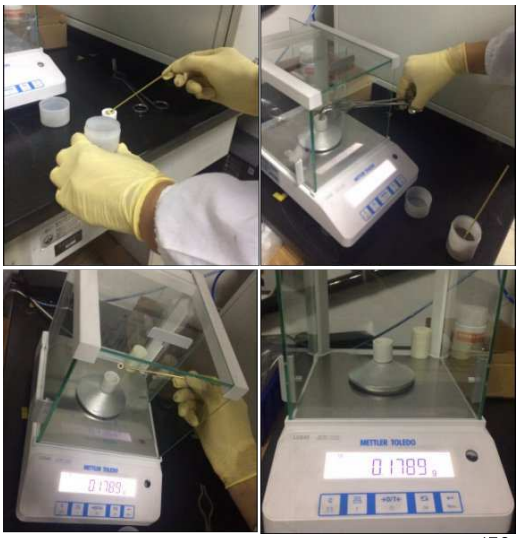
Step	Figure	Description
A	 IEC	Use a set of compressor and die to compress the dried sample, ensure the sample be compressed to a tablet and the tablet will not fall apart by holding with a pair of tweezers.
B	 IEC	Weigh about 0,1 g of the tableted sample, and transfer to a calcined crucible. Add an appropriate amount of tungsten flux particles according to the instructions of the apparatus (suggested range 1,5 g to 2,0 g) into the crucible.
C	 IEC	Place the crucible in the furnace after the samples are well blended and perform the measurement. Record the results.

Figure A.2 – A-B-C Procedures of sample preparation and carbon content determination

A.3 Data analysis for carbon content determination

A series of samples with the carbon content from 1,30 % to 100 % were carried out under repeatability conditions, i.e. one operator, same apparatus, identical operating conditions, same calibration, and a minimum period of time, each sample making three determinations of carbon content at each level. The test samples used and the results obtained are listed in Table A.1.

Table A.1 – Measurement method consistency and measurement results of different samples in the same laboratory

Sample	Sample amount	Carbon content in sample	Average value	Absolute deviation	Relative deviation	Standard deviation
	g	%	%	%	%	%
A-1	0,118 4	1,30	1,30	0	0	0,005 77
	0,138 1	1,30		0	0	
	0,110 2	1,31		0,01	0,77	
A-2	0,096 6	19,53	19,50	0,03	0,15	0,060 83
	0,096 2	19,43		−0,07	−0,36	
	0,095 6	19,54		0,04	0,21	
A-3	0,058 2	49,60	49,66	−0,06	−0,12	0,103 92
	0,055 3	49,60		−0,06	−0,12	
	0,047 7	49,78		0,12	0,24	
A-4	0,027 5	60,10	60,01	0,09	0,15	0,387 34
	0,020 1	60,35		0,34	0,57	
	0,022 5	59,59		−0,42	−0,70	
A-5	0,021 3	87,97	88,79	−0,82	−0,92	1,337 70
	0,021 8	90,34		1,55	1,75	
	0,021 2	88,08		−0,71	−0,80	
A-6	0,021 2	99,74	98,75	0,99	1,00	1,367 22
	0,010 1	97,19		−1,56	−1,58	
	0,018 5	99,32		0,57	0,58	

Conduct the carbon content determination experiment for three times and use the average value as the final result. Results from Table A.1 show that the deviation of carbon content in the same laboratory is lower than 5 %.

Two samples with the carbon content 1,20 % and 12,80 % were carried out in different laboratories under identical operating conditions. The test samples used and the results obtained are listed in Table A.2.

Table A.2 – Inter-laboratory consistency and measurement results of the same sample

Sample	Sample amount g	Experimental carbon content %	Average value %	Absolute deviation %	Relative deviation %	Standard deviation %	Data sources
B-1	0,103 7	1,21	1,21	0,00	0,41	0,008 37	Laboratory 1
	0,104 8	1,21		0,00	0,41		
	0,104 6	1,21		0,00	0,41		
	0,103 5	1,19		−0,02	−1,24		Laboratory 2
	0,101 4	1,20		−0,01	−0,41		
	0,102 8	1,21		0,00	0,41		
B-2	0,103 3	12,86	12,84	0,02	0,19	0,028 81	Laboratory 1
	0,100 9	12,86		0,02	0,19		
	0,103 2	12,84		0,00	0,04		
	0,104 2	12,85		0,01	0,12		Laboratory 2
	0,102 5	12,79		−0,05	−0,35		
	0,100 3	12,81		−0,03	−0,19		

Results from Table A.2 show that the deviation of carbon content in different laboratories is lower than 5 %.

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