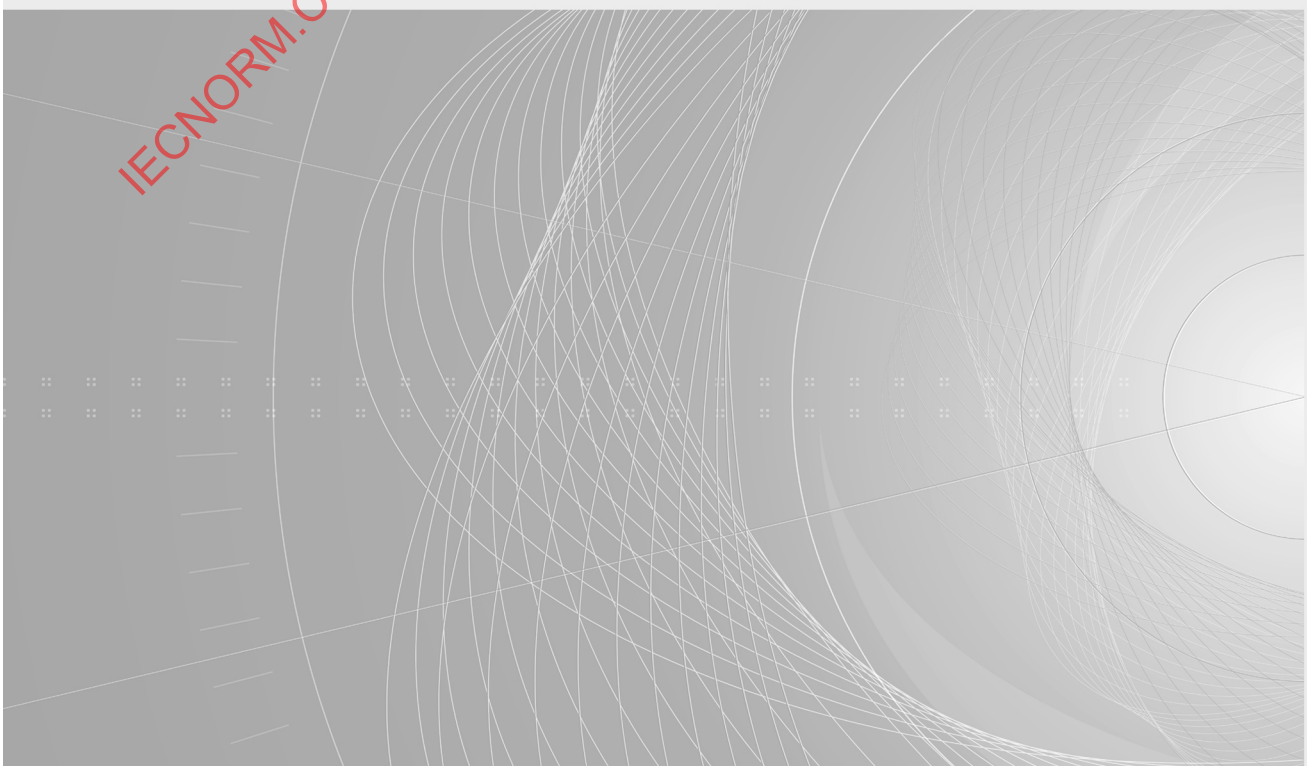
IEC TS 62607-6-19

Edition 1.0 2021-10

TECHNICAL SPECIFICATION

colour inside

Nanomanufacturing – Key control characteristics – Part 6-19: Graphene-based material – Elemental composition: CS analyser, ONH analyser



IEC TS 62607-6-19:2021-10(en)

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Nanomanufacturing – Key control characteristics – Part 6-19: Graphene-based material – Elemental composition: CS analyser, ONH analyser

INTERNATIONAL ELECTROTECHNICAL COMMISSION

ICS 07.120

ISBN 978-2-8322-1033-0

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NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 6-19: Graphene-based material – Elemental composition: CS analyser, ONH analyser

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IEC 62607-6-19 has been prepared by IEC technical committee 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
113/557/DTS	113/599/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 of the IEC TS 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- withdrawn,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.

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INTRODUCTION

In recent decades, graphene has attracted extensive attention from academy and industry, because of its extraordinary physical and chemical properties for promising applications in energy storage, electronics, composites, etc. For most graphene powder available either in the laboratory or on the market, apart from carbon, the presence of other elements (e.g. sulfur, oxygen, nitrogen, hydrogen) is inevitable in the course of graphene fabrication. Heteroatoms in graphene can change the material's energy band at different levels, thus affecting its electrical properties and thermal conductivity [1],[2][1]. Therefore, the heteroatom content is a key control characteristic which helps to ascertain the structure and purity of graphene powder, and its determination is significant for the production and application of graphene.

A method used to determine the elemental composition in graphene is the combustion/pyrolysis method, which infers the elemental composition in a sample by analysing the content of the combustion or pyrolysis gases. This method has high analysis efficiency and convenience of operation, but different instruments will provide different levels of measurement uncertainty.

In general, the combustion/pyrolysis method is established on an organic elemental analyser (EA), which uses a thermal conductivity detector (TCD) to analyse the components of the combustion or pyrolysis gases. But for graphene powder, EA is not an excellent tool to access the heteroatom content. One reason for this is that graphene has low density and sputtering happens during combustion. Another reason is that the pyrolysis temperature in EA is set at a relatively low value (e.g. 1 150 °C), which is sufficient for organics but not high enough to completely release oxygen or other atoms in graphene.

The use of a carbon/sulfur analyser (CS analyser) and an oxygen/nitrogen/hydrogen analyser (ONH analyser) can circumvent the above-mentioned problems and provide an efficient and well repeatable method for determining heteroatom content in graphene [3]. The CS analyser quantitatively analyses the combustion gas components using the infrared gas detector (IGD), while the ONH analyser quantitatively analyses the pyrolysis gas components using the TCD and IGD. The instrument has a higher pyrolysis temperature and the measurement of target gases is also completely different.

This document focuses on the determination of chemical composition in graphene powder and standardization of the procedures.

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

NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 6-19: Graphene-based material – Elemental composition: CS analyser, ONH analyser

1 Scope

This part of IEC TS 62607 establishes a standardized method to determine the chemical key control characteristic

- elemental composition

for powder consisting of graphene-based material by

- CS analyser and ONH analyser.

The method as described in this document determines the content of carbon (C), sulfur (S), oxygen (O), nitrogen (N) and hydrogen (H).

The carbon (C) and sulfur (S) content in graphene powder is derived by the content of converted CO, CO_2 and SO_2, which is determined by infrared gas detector (IGD) using a non-dispersive infrared adsorption method in CS analyser.

The content of oxygen (O), nitrogen (N) and hydrogen (H) in graphene powder is derived by ONH analyser using pyrolysis method. The O content is obtained according to the content of converted CO and CO_2, which is determined by IGD using a non-dispersive infrared adsorption method. The N content is obtained according to the content of converted N_2, which is determined by a thermal conductivity detector (TCD) method. The H content is obtained by measuring converted H_2 or H_2O, corresponding to TCD or IGD method.

- The method is applicable for graphene, graphene oxide (GO) and reduced graphene oxide (rGO) in powder form.

2 Normative references

There are no normative references in this document.

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
two-dimensional material
2D material
material, consisting of one or several layers with the atoms in each layer strongly bonded to neighbouring atoms in the same layer, which has one dimension, its thickness, in the nanoscale or smaller and the other two dimensions generally at larger scales

Note 1 to entry: The number of layers when a two-dimensional material becomes a bulk material varies depending on both the material being measured and its properties. In the case of graphene layers, it is a two-dimensional material up to 10 layers thick for electrical measurements, beyond which the electrical properties of the material are not distinct from those for the bulk [also known as graphite].

Note 2 to entry: Interlayer bonding is distinct from and weaker than intralayer bonding.

Note 3 to entry: Each layer may contain more than one element.

Note 4 to entry: A two-dimensional material can be a nanoplate.

[SOURCE: ISO/TS 80004-13:2017, 3.1.1.1]

3.1.2
graphene
graphene layer
single-layer graphene
monolayer graphene
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-layer 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]

3.1.3
graphene-based material
GBM
graphene material
grouping of carbon-based 2D materials that include one or more of graphene, bilayer graphene, few-layer graphene, graphene nanoplate, and functionalized variations thereof as well as graphene oxide and reduced graphene oxide.

Note 1 to entry: "Graphene material" is a short name for graphene-based material.

3.1.4
graphene oxide
GO
chemically modified graphene prepared by oxidation and exfoliation of graphite, causing extensive oxidative modification of the basal plane

Note 1 to entry: Graphene oxide is a single-layer material with a high oxygen content, typically characterized by C/O atomic ratios of approximately 2,0 depending on the method of synthesis.

[SOURCE: ISO/TS 80004-13:2017, 3.1.2.13]

3.1.5
reduced graphene oxide
rGO
reduced oxygen content form of graphene oxide

Note 1 to entry: This can be produced by chemical, thermal, microwave, photo-chemical, photo-thermal or microbial/bacterial methods or by exfoliating reduced graphite oxide.

Note 2 to entry: If graphene oxide was fully reduced, then graphene would be the product. However, in practice, some oxygen containing functional groups will remain and not all sp^3 bonds will return back to sp^2 configuration. Different reducing agents will lead to different carbon to oxygen ratios and different chemical compositions in reduced graphene oxide.

Note 3 to entry: It can take the form of several morphological variations such as platelets and worm-like structures.

[SOURCE: ISO/TS 80004-13:2017, 3.1.2.14]

3.1.6
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.

3.1.7
sectional blank detail specification
SBDS
specification based on a blank detail specification adapted for a subgroup of the nano-enabled product

Note 1 to entry: In general the sectional blank detail specification contains a subset of those key control characteristics (KCCs) listed in the blank detail specification. In addition, sectional specific KCCs may be added if they are not listed in the blank detail specification.

Note 2 to entry: The templates defined in the sectional blank detail specification may contain KCCs with and without assigned values and attributes.

Note 3 to entry: The section can be defined by application, manufacturing method or general material properties.

3.1.8
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. 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. Standards Development Organizations 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.

3.1.9
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.

3.2 Key control characteristics measured according to this document

3.2.1
carbon content
<2D material> amount of total carbon in the 2D material

3.2.2
sulfur content
<2D material> amount of total sulfur in the 2D material

3.2.3
oxygen content
<2D material> amount of total oxygen in the 2D material

[SOURCE: ISO/TS 80004-13:2017, 3.4.2.7]

3.2.4
nitrogen content
<2D material> amount of total nitrogen in the 2D material

3.2.5
hydrogen content
<2D material> amount of total hydrogen in the 2D material

3.3 Terms related to the measurement method

3.3.1
carbon/sulfur analyser
CS analyser
analyser for the determination of carbon and sulfur content in metallic mineral, ceramic, cement, lime, rubber, coal, coke, refractory materials, carbide, graphite, oil products, catalysts, soil and other solid materials

3.3.2
oxygen/nitrogen/hydrogen analyser
ONH analyser
analyser for the determination of oxygen, nitrogen and hydrogen content in ferrous and non-ferrous metals, rare earth materials, and some other inorganic materials

3.3.3
infrared detector
IR detector
flame detector responding only to radiation having wavelengths greater than 850 nm

[SOURCE: ISO 7240-10:2012, 3.2]

3.3.4
thermal conductivity detector
TCD
detector that measures the difference in thermal conductivity between two gas streams when a sample (gas mixture) passes through the sample channel

Note 1 to entry: The TCD is a dual channel detector, requiring a reference flow of pure carrier gas through the reference channel.

Note 2 to entry: The use of helium, argon or hydrogen is recommended as carrier gas except when the sample contains either of these two substances to be measured.

Note 3 to entry: The detector consists of a bridge circuit; the change in resistance in the sample channel during the passage of the sample produces an out-of-balance signal that is the basis of the detection. The detector responds to all components except the carrier gas and it is non-destructive.

[SOURCE: ISO 14532:2014, 2.4.9]

4 General

4.1 Measurement principle

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 combustion gases pass through a dust filter and moisture absorber for purification. The carbon content or sulfur content is detected by IGD using infrared absorption method. Infrared cells can be adapted according to user's requirements.

The ONH analyser is based on the impulse heating inert gas fusion (IGF) 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 [4], the nitrogen content to elemental N_2, and the hydrogen content to H_2. All CO, H_2 and N_2 are transported by carrier gas to the oxidation furnace where copper oxide converts CO to CO_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, which senses differences in the thermal conductivity between target gas and the reference carrier gas [5]. 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.

4.2 Sample preparation method

Graphene powder shall be dried with desiccant prior to use to remove residual moisture. After drying to a constant mass, store the samples in a desiccator for use.

For CS content measurement, in order to avoid sample splatter during combustion, graphene powder with a mass of at least 20 mg shall be pressed with pelletizer under 3 MPa to 5 MPa within 1 min. In order to avoid the influence of residual carbon, the test corundum crucible needs to be burned at 1 000 °C for 4 h, cooling down naturally and being stored in a desiccator for use, unless otherwise stated.

For ONH content measurement, in order to minimize the effect of the oxygen absorption, graphene powder with a mass of at least 20 mg shall be pressed with pelletizer under 10 MPa to 15 MPa within 1 min. Nickel foils are used as combustion improver. Cut the nickel foils with scissors to a certain size (e.g. 2 cm × 2 cm), wash the foils with solvents (e.g. ethanol absolute or acetone) and dry with air blower. Then encapsulate graphene powder with nickel foils for weighing.

4.3 Description of measurement equipment / apparatus

A CS analyser can simultaneously determine the content of carbon and sulfur in samples. It includes combustion system, automatic sample loading system, infrared detecting system and application software. In the combustion system, reliable and durable heating elements and combustion tube are adopted to achieve the stability of the heating rate and the accuracy of the temperature which is set according to the sample. Oxygen supplied steadily from both the gun and the chamber ensures complete combustion of the sample. An integrated automatic sample loading system can realize the batch analyses. The infrared system can be customized in a flexible way, and several independent physical channels can be assembled. As for the automatic analysis process, 60 samples can be stored at one time.

An ONH analyser can measure the content of oxygen, nitrogen and hydrogen in samples. A solid state infrared detector is used for oxygen measurement. A thermal conductivity detector is used for nitrogen measurement. Both thermal conductivity detector and infrared detector can be used for hydrogen measurement. The length of IR cells can be customized according to the content of samples. The pulse heating furnace ensures a high temperature (maximum 3 000 °C) in a short time.

For sample weighing, an electronic balance which has a capacity of 120 g with precision accuracy of 0,1 mg is needed.

4.4 Supporting materials

During the analysis, reagents and materials are needed. Unless otherwise stated, use only reagents of recognized analytical grade.

4.4.1 **helium gas, argon gas, oxygen gas and nitrogen gas**, high purity, total impurity content 0,000 5 % (m/m).

4.4.2 **corundum crucible**, single use, high purity, suitable for use with the apparatus.

4.4.3 **graphite crucible**, single use, high purity, suitable for use with the apparatus.

4.4.4 **crucible tongs**, for handling the crucibles used.

4.4.5 **nickel foils**, suitable for sample encapsulation, fusion and catalyser.

4.4.6 **carbon dioxide absorbent**, suitable for filtering carrier gas.

4.4.7 **magnesium perchlorate, $Mg(ClO_4)_2$**, particle size from 1,2 mm to 2,0 mm, or anhydrous calcium sulfate, particle size from 0,6 mm to 0,85 mm.

4.4.8 **copper (II) oxide**, particle size from 1,2 mm to 2,0 mm, or anhydrous calcium sulfate, particle size from 0,6 mm to 0,85 mm. Used for oxidation catalyst.

4.4.9 degreasing cotton, suitable for filtering dust due to high-temperature heating in the impulse furnace.

4.4.10 appropriate solvent, suitable for washing nickel foils or crucible tongs, e.g. ethanol absolute or acetone.

4.4.11 certified reference materials, anthracite for C and S content measurement, iron powder for O content measurement, and steel for N and H content measurement.

4.4.12 scissors, air blower and tweezers, scissors used to cut nickel foils and air blower used for drying nickel foils washed with solvents.

4.5 Ambient conditions during measurement

All measurements shall be carried out at room temperature and relative humidity below 70 %. Specific temperature and relative humidity are not required.

5 Measurement procedure

5.1 Calibration of measurement equipment

Before starting the measurements, the equipment shall be calibrated according to the manufacturer's requirements and instructions. This can be achieved by measuring chemical compositions in certified reference materials.

5.2 Detailed protocol of the measurement procedure

After the instrument is turned on, the baseline fluctuation is observed in the release curve area of the main interface. After the baseline is stable, the fluctuation is within 30 mV and the analysis operation can be performed. Use the certified reference materials to adjust the calibration curve of the instrument and match it with the standard value to obtain a suitable calibration curve. When the analysis results meet the requirements, the actual sample analysis can be performed.

For CS content measurement, use a corundum crucible to load the sample. After weighing the sample in a crucible, the mass is transferred from the interfaced balance to the computer. If required, sample mass can also be entered manually. After having placed the crucible on the pedestal, the analysis starts. The transmission device will send the corundum crucible into the furnace chamber of the CS analyser, and then it will automatically perform operations such as degassing, gas displacement, and burning. The detector signals and instrument parameters are displayed during analysis. The typical analysis time is 60 s to 180 s for CS analyser. Evaluation of the signals and display of the results are done automatically.

For ONH content measurement, nickel foils are used as combustion improver and sample container. Weigh nickel foils before and afterwards, determining the net mass of graphene sample encapsulated by nickel foils. The mass is transferred from the interfaced balance to the PC. If required, sample mass can also be entered manually. Place a graphite crucible on the pedestal of the furnace chamber of the ONH analyser, and the command is entered by the control system. Afterwards, the pedestal and chamber are locked, and the analysis starts. ONH analyser will automatically perform operations such as degassing, gas displacement, and burning. The detector signals and instrument parameters are displayed during analysis. The typical analysis time is 180 s for ONH analyser. Evaluation of the signals and display of the results are done automatically.

Finally, record the analysis results and end the test.

WARNING – The risks involved when using an apparatus for fusing the test samples are mainly risks of burns. It is therefore essential to use crucible tongs and appropriate containers for the used crucibles.

NOTE 1 Each item of equipment is left to stabilize for the time recommended by the equipment manufacturers when the main supply is switched on after being out of action for any length of time.

NOTE 2 After cleaning the furnace chamber and/or changing filters after the equipment has been inoperative for a period, the equipment is stabilized by burning several samples of similar type to the samples to be analysed prior to setting up for analysis.

5.3 Measurement accuracy

To obtain reliable data, the effectiveness of the installed reagents shall be checked before measurements. For instance, the normal shape of $Mg(ClO_4)_2$ appears like white flakes. It will agglomerate and fail after water absorption, and should be replaced. For sample weighing, an electronic balance which has a capacity of 120 g with precision accuracy of 0,1 mg shall be used.

6 Data analysis / interpretation of results

The measurement signals will be outputted to the data acquisition board of the computer. The mass fraction of the measured elements is finally obtained by acquisition, processing, integration and operation of the computer software. Mean value, standard deviation and relative standard deviation of the data can be calculated by the software.

7 Results to be reported

7.1 General

The results of the measurement shall be documented in a measurement report, including the date and time of the measurement as well as the name and signature of the person responsible for the accuracy of the report. Guidelines are given in Annex A.

7.2 Product / sample identification

The report shall contain all information to identify the test sample and trace back the history of the sample.

a) General procurement information.

b) General material description

7.3 Test conditions

The laboratory ambient conditions during the test.

– Temperature range: 17 °C < T < 30 °C.

– Range of relative humidity: 40 % < RH < 70 %.

7.4 Measurement specific information

– The instruments used, including the manufacturer, the name, model, etc.

– The certified reference material used.

– Calibration status of equipment.

– Any unusual features noted during the measurements.

– Any optional operation which may have influenced the results.

7.5 Test results

– All information necessary for the identification of the sample and the date of the analysis.

– Results of elemental composition (C/S/O/N/H) measured in accordance with this document. See Annex B for detailed information.

Annex A
(informative)

Test report

A.1 Recommended format of the test report

The form of the test report shall be oriented on the relevant material specification (e.g. IEC TS 62565-3-1, a related sectional blank detail specification or detail specification. Table A.1, Table A.2, Table A.3 and Table A.4 are guidelines to write the test report and can be customized to fulfil the requirements of the involved parties.

Table A.1 – Product identification

Item No	Item		Information
1.1	Supplier		
1.2	Trade name		
1.3	ID number		
1.4	Typical batch quantity	Mass [g]	
1.5	Traceability requirements	☐ Batch number ☐ Serial number ☐ Others, specify ……	
		Manufacturing date	
1.6	Specification	Number	
		Revision level	
		Date of issue	
1.7	Material Safety Data Sheet (MSDS) available	☐ No	
		☐ Yes Reference	

Table A.2 – General material description

Item No	Item	Information
2.1	Material type	
2.2	Manufacturing method	
2.3	Physical form	
2.5	Shelf life	
2.6	Typical batch size	

Table A.3 – Information relating to test

Item No	Item		Information
3.1	Instruments	Name	
		Manufacturer	
		Model	
3.2	Certified reference material		
3.3	Hydrogen measurement method		
3.4	Combustion improver		
3.5	Solvents		
3.6	Carrier gas		
3.7	Number of measurements		

Table A.4 – Measurement results

Measurement	Mass fraction of C (%)	Mass fraction of S (%)	Mass fraction of O (%)	Mass fraction of N (%)	Mass fraction of H (%)
1					
2					
3					
4					
5					
6					
Average (mass fraction (%))					
Standard deviation					

Annex B
(informative)

Case study: Comparative results between CS/ONH analyser and EA

B.1 Measurement sample

Different types of samples are used for measurement, including graphene powder (G,p), graphene dispersion (G,d), graphene oxide powder (GO,p), graphene oxide film (GO,f), graphene oxide dispersion (GO,d), and reduced graphene oxide powder (RGO,p).

B.2 Measurement equipment

CS analyser and ONH analyser are used to measure the content of C/S and O/N, respectively. Elemental analyser (EA) is used for comparative measurements.

B.3 Measurement results

B.3.1 General

Samples with high/low C/S/O/N content are used for measurement. Each experiment is repeated at least three times for reproducibility.

B.3.2 Measuring samples with low C content (mass fraction (%)):

Table B.1 and Figure B.1 compare the measurement results of samples with low C content by CS analyser and EA.

Table B.1 – Measurement results of samples with low C content

	Measurements with CS analyser						Measurements with EA					
	1	2	3	4	mean	SD	1	2	3	4	mean	SD
GO,f	40,370	40,414	40,756	41,550	40,773	0,546	50,500	50,780	50,350	49,840	50,368	0,394
GO,d	36,659	37,784	36,438	36,601	36,871	0,616	49,340	48,750	49,100	49,150	49,085	0,246
GO,p	43,637	42,308	43,805	43,051	43,200	0,677	45,040	45,040	45,580	45,930	45,398	0,437

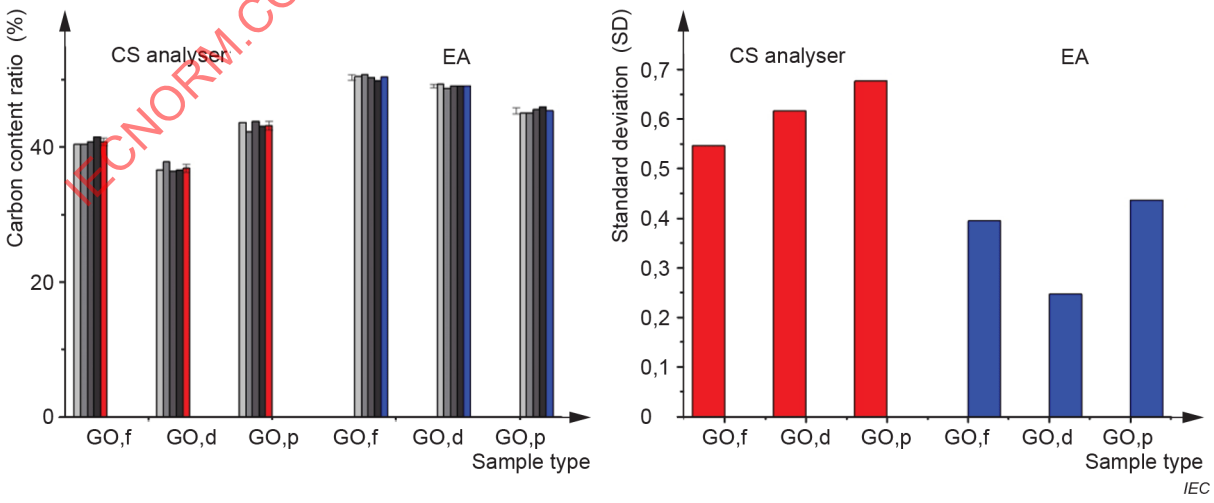

Figure B.1 – Measurement results of samples with low C content

B.3.3 Measuring samples with high C content (mass fraction (%)):

Table B.2 and Figure B.2 compare the measurement results of samples with high C content by CS analyser and EA.

Table B.2 – Measurement results of samples with high C content

Measurements with CS analyser						Measurements with EA				
	1	2	3	mean	SD	1	2	3	mean	SD
G,p	92,513	93,196	93,527	93,079	0,517	99,210	99,230	99,610	99,350	0,225
G,d	90,838	91,484	91,534	91,285	0,388	88,410	87,170	86,790	87,457	0,847
RGO,p	88,906	89,062	88,355	88,774	0,371	87,480	85,710	87,510	86,900	1,031

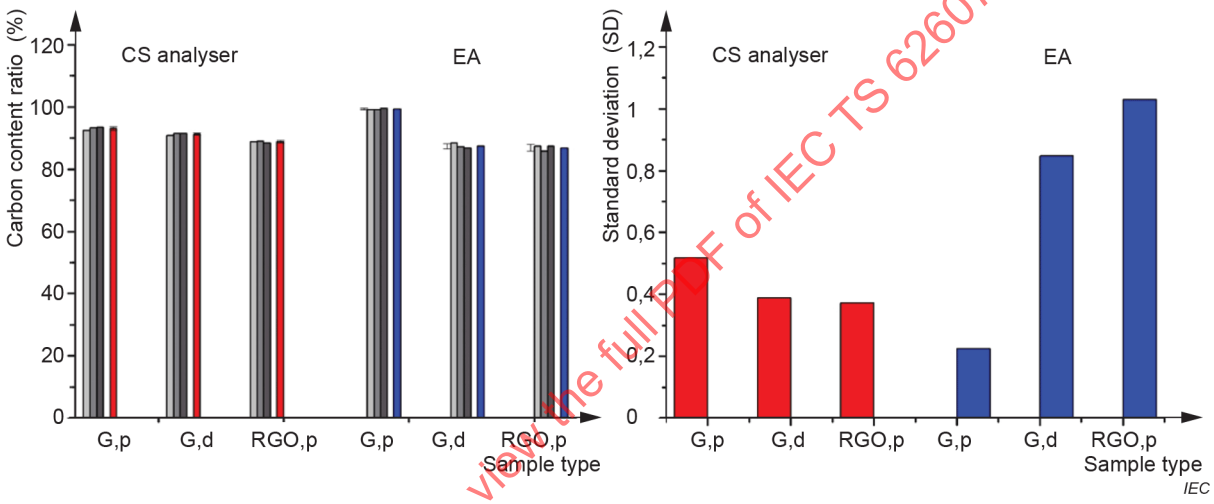

Figure B.2 – Measurement results of samples with high C content

B.3.4 Measuring samples with low S content (mass fraction (%)):

Table B.3 and Figure B.3 compare the measurement results of samples with low S content by CS analyser and EA.

Table B.3 – Measurement results of samples with low S content

Measurements with CS analyser							Measurements with EA					
	1	2	3	4	mean	SD	1	2	3	4	mean	SD
GO,f	1,755	1,829	1,914	1,802	1,825	0,067	0,917	0,940	0,942	1,018	0,954	0,044
GO,d	2,162	1,937	2,139	1,824	2,016	0,163	1,125	1,101	1,181	1,271	1,170	0,076
GO,p	1,743	1,822	1,687	1,752	1,751	0,055	9,685	8,352	5,418	5,423	7,220	2,147

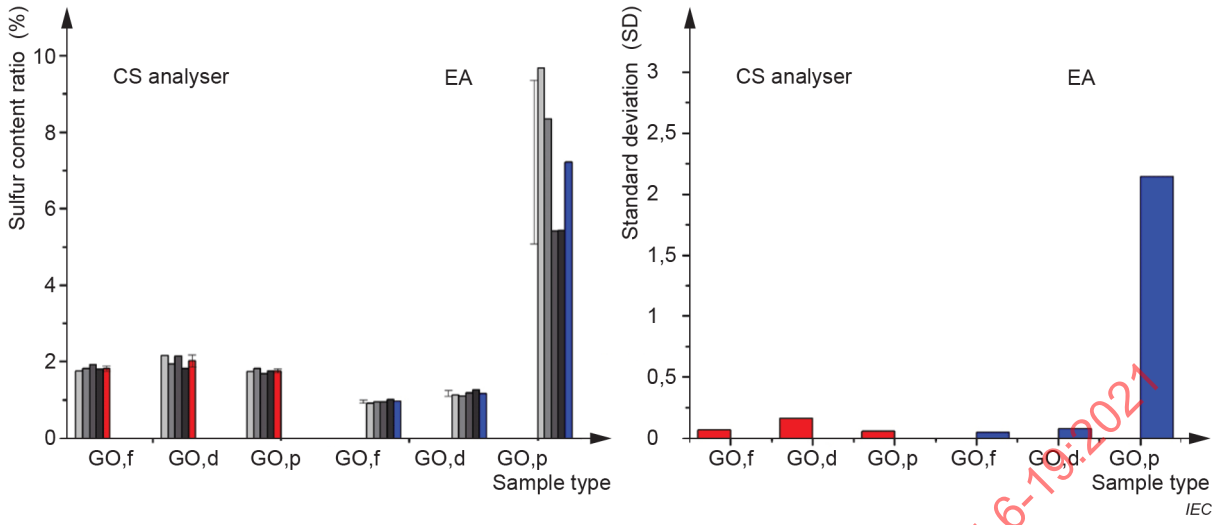

Figure B.3 – Measurement results of samples with low S content

B.3.5 Measuring samples with high S content (mass fraction (%)):

Table B.4 and Figure B.4 compare the measurement results of samples with high S content by CS analyser and EA.

Table B.4 – Measurement results of samples with high S content

Measurements with CS analyser						Measurements with EA				
	1	2	3	mean	SD	1	2	3	mean	SD
G,p	1,962	2,072	2,081	2,038	0,066	0,910	0,889	0,711	0,837	0,109
G,d	2,317	2,377	2,368	2,354	0,032	3,341	3,440	3,557	3,446	0,108
RGO,p	3,383	3,483	3,373	3,413	0,061	2,076	1,769	1,775	1,873	0,176

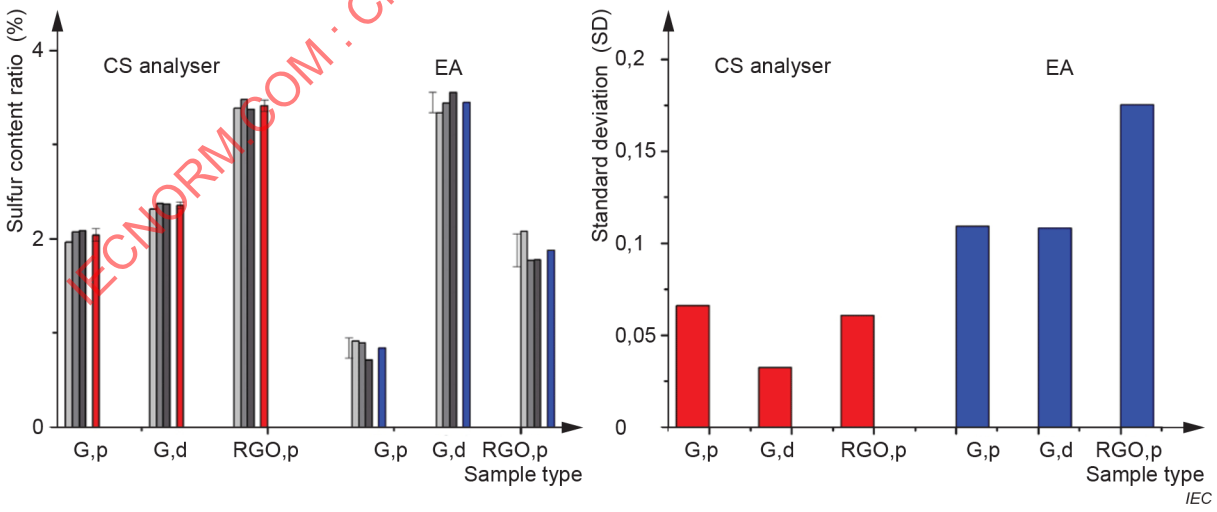

Figure B.4 – Measurement results of samples with high S content

B.3.6 Measuring samples with low O content (mass fraction (%)):

Table B.5 and Figure B.5 compare the measurement results of samples with low O content by ONH analyser and EA.

Table B.5 – Measurement results of samples with low O content

Measurements with ONH analyser						Measurements with EA				
	1	2	3	mean	SD	1	2	3	mean	SD
G,p	2,713	2,773	3,037	2,841	0,172	0,305	0,039	0,582	0,309	0,272
G,d	3,666	3,420	3,663	3,583	0,141	8,274	6,580	7,211	7,355	0,856
RGO,p	5,478	5,254	5,418	5,383	0,116	6,255	5,884	3,328	5,156	1,594

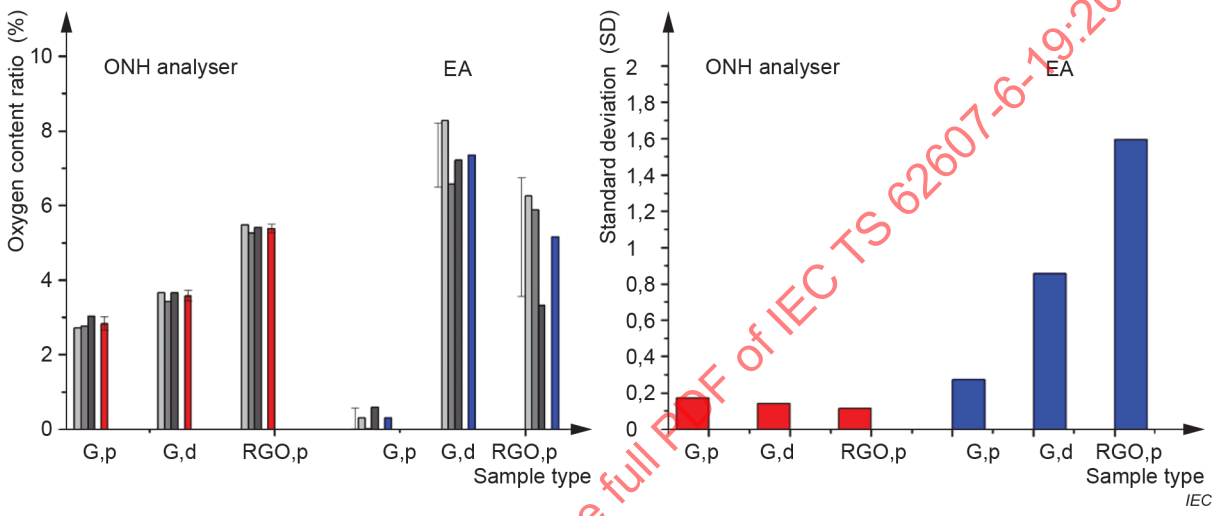

Figure B.5 – Measurement results of samples with low O content

B.3.7 Measuring samples with high O content (mass fraction (%)):

Table B.6 and Figure B.6 compare the measurement results of samples with high O content by ONH analyser and EA.

Table B.6 – Measurement results of samples with high O content

Measurements with ONH analyser							Measurements with EA					
	1	2	3	4	mean	SD	1	2	3	4	mean	SD
GO,f	40,495	40,089	39,508	39,823	39,979	0,418	47,090	39,149	41,381	45,675	43,324	3,693
GO,d	37,489	39,232	40,242	38,910	38,968	1,138	46,632	45,503	41,082	40,312	43,382	3,150
GO,p	39,305	39,715	39,898	39,813	39,683	0,263	47,090	39,149	41,381	45,675	43,324	3,693

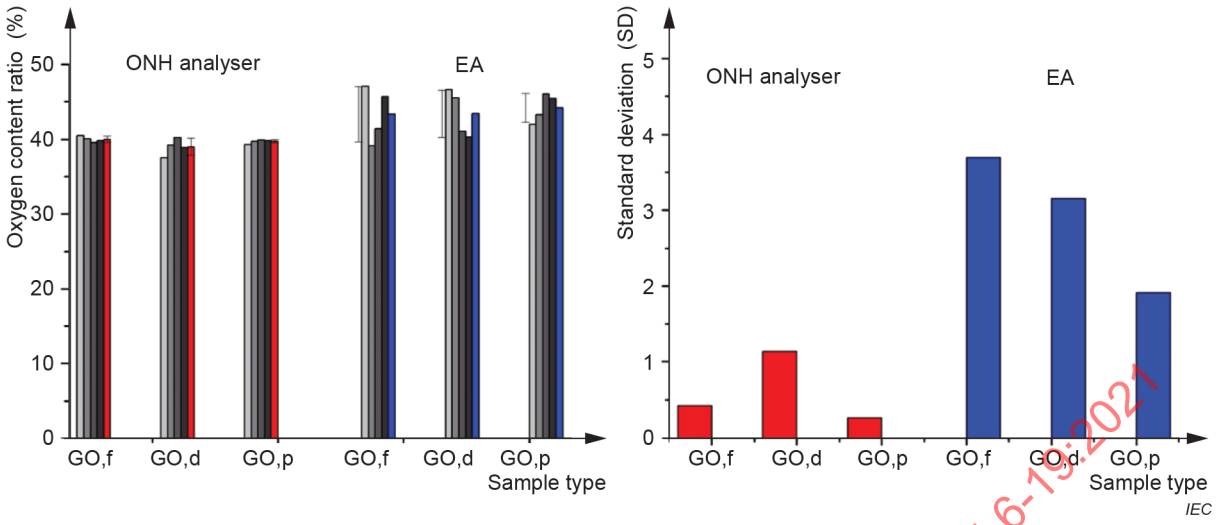

Figure B.6 – Measurement results of samples with high O content

B.3.8 Measuring samples with low N content (mass fraction (%)):

Table B.7 and Figure B.7 compare the measurement results of samples with low N content by ONH analyser and EA.

Table B.7 – Measurement results of samples with low N content

	Measurements with ONH analyser						Measurements with EA					
	1	2	3	4	mean	SD	1	2	3	4	mean	SD
GO,f	0,159	0,181	0,153	0,163	0,164	0,012	0,860	0,460	0,610	0,690	0,655	0,167
GO,d	0,226	0,200	0,200	0,197	0,206	0,014	0,390	0,680	0,730	0,570	0,593	0,151
GO,p	0,149	0,151	0,142	0,153	0,149	0,005	0,600	0,630	0,490	0,540	0,565	0,062

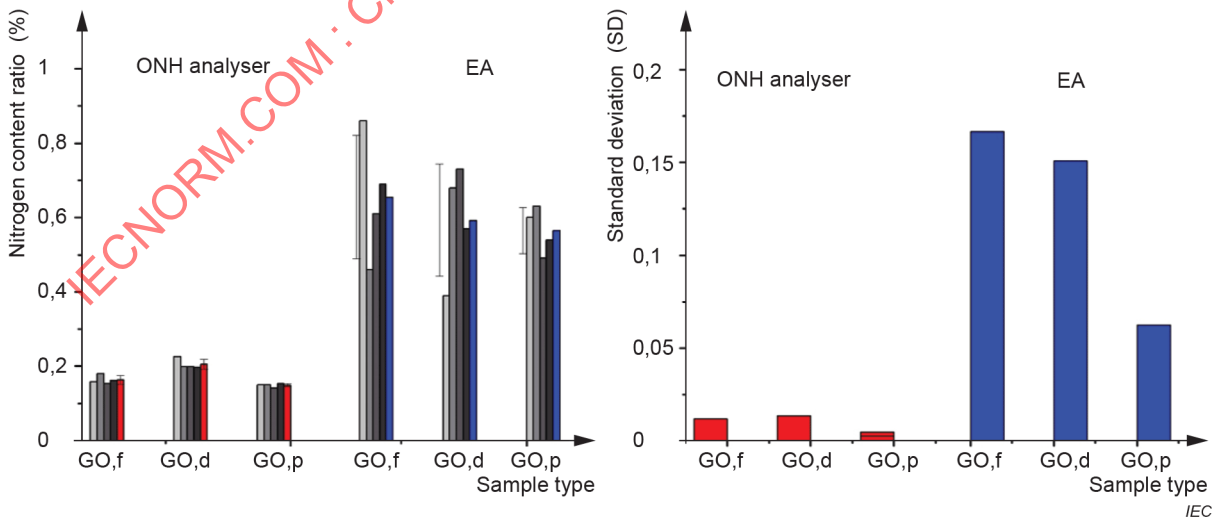

Figure B.7 – Measurement results of samples with low N content

B.3.9 Measuring samples with high N content (mass fraction (%)):

Table B.8 and Figure B.8 compare the measurement results of samples with high N content by ONH analyser and EA.