

# TECHNICAL SPECIFICATION



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



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

INTERNATIONAL  
ELECTROTECHNICAL  
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## CONTENTS

FOREWORD.....	4
INTRODUCTION.....	6
1 Scope.....	7
2 Normative references .....	7
3 Terms and definitions .....	7
3.1 General terms .....	7
3.2 Key control characteristics measured according to this document .....	9
3.3 Terms related to the measurement method .....	10
4 General .....	10
4.1 Measurement principle.....	10
4.2 Sample preparation method .....	11
4.3 Description of measurement equipment / apparatus .....	11
4.4 Supporting materials .....	11
4.5 Ambient conditions during measurement.....	12
5 Measurement procedure .....	12
5.1 Calibration of measurement equipment .....	12
5.2 Detailed protocol of the measurement procedure .....	12
5.3 Measurement accuracy .....	13
6 Data analysis / interpretation of results.....	13
7 Results to be reported .....	13
7.1 General.....	13
7.2 Product / sample identification .....	13
7.3 Test conditions .....	13
7.4 Measurement specific information.....	13
7.5 Test results .....	13
Annex A (informative) Test report .....	14
A.1 Recommended format of the test report .....	14
Annex B (informative) Case study: Comparative results between CS/ONH analyser and EA .....	16
B.1 Measurement sample.....	16
B.2 Measurement equipment.....	16
B.3 Measurement results.....	16
B.3.1 General .....	16
B.3.2 Measuring samples with low C content (mass fraction (%)):	16
B.3.3 Measuring samples with high C content (mass fraction (%)):	17
B.3.4 Measuring samples with low S content (mass fraction (%)):	17
B.3.5 Measuring samples with high S content (mass fraction (%)):	18
B.3.6 Measuring samples with low O content (mass fraction (%)):	18
B.3.7 Measuring samples with high O content (mass fraction (%)):	19
B.3.8 Measuring samples with low N content (mass fraction (%)):	20
B.3.9 Measuring samples with high N content (mass fraction (%)):	20
Bibliography.....	23
Figure B.1 – Measurement results of samples with low C content .....	16
Figure B.2 – Measurement results of samples with high C content .....	17

Figure B.3 – Measurement results of samples with low S content.....	18
Figure B.4 – Measurement results of samples with high S content .....	18
Figure B.5 – Measurement results of samples with low O content .....	19
Figure B.6 – Measurement results of samples with high O content.....	20
Figure B.7 – Measurement results of samples with low N content .....	20
Figure B.8 – Measurement results of samples with high N content .....	21
Figure B.9 – A summary of SD of all measurements .....	22
Table A.1 – Product identification .....	14
Table A.2 – General material description .....	14
Table A.3 – Information relating to test .....	15
Table A.4 – Measurement results.....	15
Table B.1 – Measurement results of samples with low C content.....	16
Table B.2 – Measurement results of samples with high C content .....	17
Table B.3 – Measurement results of samples with low S content.....	17
Table B.4 – Measurement results of samples with high S content .....	18
Table B.5 – Measurement results of samples with low O content .....	19
Table B.6 – Measurement results of samples with high O content.....	19
Table B.7 – Measurement results of samples with low N content.....	20
Table B.8 – Measurement results of samples with high N content .....	21

## INTERNATIONAL ELECTROTECHNICAL COMMISSION

**NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –****Part 6-19: Graphene-based material –  
Elemental composition: CS analyser, ONH analyser**

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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](http://www.iec.ch/members_experts/refdocs). The main document types developed by IEC are described in greater detail at [www.iec.ch/standardsdev/publications](http://www.iec.ch/standardsdev/publications).

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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]<sup>1</sup>. 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.

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<sup>1</sup> 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<sub>2</sub> and SO<sub>2</sub>, 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<sub>2</sub>, which is determined by IGD using a non-dispersive infrared adsorption method. The N content is obtained according to the content of converted N<sub>2</sub>, which is determined by a thermal conductivity detector (TCD) method. The H content is obtained by measuring converted H<sub>2</sub> or H<sub>2</sub>O, 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.