

TECHNICAL SPECIFICATION



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

INTERNATIONAL
ELECTROTECHNICAL
COMMISSION

ICS 07.120

ISBN 978-2-8322-4377-0

Warning! Make sure that you obtained this publication from an authorized distributor.

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**

FOREWORD

- 1) The International Electrotechnical Commission (IEC) is a worldwide organization for standardization comprising all national electrotechnical committees (IEC National Committees). The object of IEC is to promote international co-operation on all questions concerning standardization in the electrical and electronic fields. To this end and in addition to other activities, IEC publishes International Standards, Technical Specifications, Technical Reports, Publicly Available Specifications (PAS) and Guides (hereafter referred to as "IEC Publication(s)"). Their preparation is entrusted to technical committees; any IEC National Committee interested in the subject dealt with may participate in this preparatory work. International, governmental and non-governmental organizations liaising with the IEC also participate in this preparation. IEC collaborates closely with the International Organization for Standardization (ISO) in accordance with conditions determined by agreement between the two organizations.
- 2) The formal decisions or agreements of IEC on technical matters express, as nearly as possible, an international consensus of opinion on the relevant subjects since each technical committee has representation from all interested IEC National Committees.
- 3) IEC Publications have the form of recommendations for international use and are accepted by IEC National Committees in that sense. While all reasonable efforts are made to ensure that the technical content of IEC Publications is accurate, IEC cannot be held responsible for the way in which they are used or for any misinterpretation by any end user.
- 4) In order to promote international uniformity, IEC National Committees undertake to apply IEC Publications transparently to the maximum extent possible in their national and regional publications. Any divergence between any IEC Publication and the corresponding national or regional publication shall be clearly indicated in the latter.
- 5) IEC itself does not provide any attestation of conformity. Independent certification bodies provide conformity assessment services and, in some areas, access to IEC marks of conformity. IEC is not responsible for any services carried out by independent certification bodies.
- 6) All users should ensure that they have the latest edition of this publication.
- 7) No liability shall attach to IEC or its directors, employees, servants or agents including individual experts and members of its technical committees and IEC National Committees for any personal injury, property damage or other damage of any nature whatsoever, whether direct or indirect, or for costs (including legal fees) and expenses arising out of the publication, use of, or reliance upon, this IEC Publication or any other IEC Publications.
- 8) Attention is drawn to the Normative references cited in this publication. Use of the referenced publications is indispensable for the correct application of this publication.
- 9) Attention is drawn to the possibility that some of the elements of this IEC Publication may be the subject of patent rights. IEC shall not be held responsible for identifying any or all such patent rights.

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.

IMPORTANT – The "colour inside" logo on the cover page of this document indicates that it contains colours which are considered to be useful for the correct understanding of its contents. Users should therefore print this document using a colour printer.

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, CO₂ and SO₂, 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 CO₂, which is determined by IGD using a non-dispersive infrared adsorption method. The N content is obtained according to the content of converted N₂, which is determined by a thermal conductivity detector (TCD) method. The H content is obtained by measuring converted H₂ or H₂O, 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.