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TECHNICAL SPECIFICATION

colour inside

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

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CONTENTS

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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- withdrawn,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