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TECHNICAL SPECIFICATION

Nanomanufacturing – Key control characteristics –
Part 6-20: Graphene-based material – Metallic impurity content: Inductively coupled plasma mass spectrometry

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IEC TS 62607-6-20 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
113/609/DTS	113/629/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 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- withdrawn,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

Graphene-based materials have wide potential applications because of their unique electrical, thermal and mechanical properties, especially in the electronics industry: batteries, integrated circuits, high-frequency electronics, displays, etc. [1], [2], [3][1]. As industry uptake on graphene-based materials increases, international standardization is critical to enable the commercialization of graphene-based materials and related products. Metal impurities within graphene-based materials have significant impact on the electrical performance in the process of industrial application. Considering the multiple production routes and producers of graphene-based materials, in order to maintain product quality and reach a consensus between the supplier and the customer, there is no doubt that accurate, reliable and reproducible measurement methods for the key parameters of graphene-based materials need to be established.

Inductively coupled plasma mass spectrometry (ICP-MS) can carry out accurate detection of trace amounts of a variety of metal impurities simultaneously, obtain species and content of each metal impurity in graphene-based materials.

The purpose of this document is to enable accurate and quantitative determination of metal impurities using ICP-MS [4], through providing optimized digestion operation, preparation procedures for graphene-based materials in powder form, measurement method and data analysis. A similar document was published as ISO/TS 13278 for carbon nanotubes (CNTs) [5]; however, it is not suitable for graphene powder because of the noticeable difference between CNTs and graphene powder, especially in terms of sample preparation (including digestion technique and digestion procedure), the properties of test samples (many more species and much wider range of content of metal impurities in graphene powder), measurement procedure and so on. Therefore, this document has been developed for graphene powder; it is based on study in VAMAS Technical Working Area 41 (TWA 41).

1 Numbers in square brackets refer to the Bibliography.

NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 6-20: Graphene-based material – Metallic impurity content: Inductively coupled plasma mass spectrometry
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1 Scope

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

– metallic impurity content

for powders of graphene-based materials by

– inductively coupled plasma mass spectrometry (ICP-MS).

The metallic impurity content is derived by the signal intensity of measured elements through MS spectrum of ICP-MS.

– The method is applicable for powder of graphene and related materials, including bilayer graphene (2LG), trilayer graphene (3LG), few-layer graphene (FLG), reduced graphene oxide (rGO) and graphene oxide (GO).
– The typical application area is in the microelectronics industry, e.g. conductive pastes, displays, etc., for manufacturers to guide material design, and for downstream users to select suitable products.

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
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 [6], 3.1.2.1]

3.1.2
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.

3.1.3
bilayer graphene
2LG
two-dimensional material consisting of two well-defined stacked graphene layers

Note 1 to entry: If the stacking registry is known, it can be specified separately, for example, as "Bernal stacked bilayer graphene".

[SOURCE: ISO/TS 80004-13:2017 [6], 3.1.2.6]

3.1.4
trilayer graphene
3LG
two-dimensional material consisting of three well-defined stacked graphene layers

Note 1 to entry: If the stacking registry is known, it can be specified separately, for example, as "twisted trilayer graphene".

[SOURCE: ISO/TS 80004-13:2017 [6], 3.1.2.9]

3.1.5
few-layer graphene
FLG
two-dimensional material consisting of three to ten well-defined stacked graphene layers

[SOURCE: ISO/TS 80004-13:2017 [6], 3.1.2.10]

3.1.6
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 [6], 3.1.2.13]

3.1.7
reduced graphene oxide
rGO
reduced oxygen content form of graphene oxide

Note 1 to entry: rGO can be produced by chemical, thermal, microwave, photo-chemical, photo-thermal, microbial or 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 [6], 3.1.2.14]

3.2 Key control characteristics measured in accordance with this document

3.2.1
metallic impurity
metallic element present in graphene-based materials and not in the crystalline structure of graphene

Note 1 to entry: The content of most metallic impurities in graphene-based materials is usually trace, but for industrial products of graphene powder, the content of a few metal impurities can be higher, e.g. Na element coming from water used in rGO production, and Cu or Fe element coming from manufacturing equipment used in the production of FLG powder.

4 General

4.1 Chemical reagents

All reagents should be guaranteed reagents (GRs) at least, and the purity quotient should be no less than 99,99 %.

4.1.1 Ultra-pure nitric acid (HNO_3, MOS (metal-oxide-semiconductor) or higher, 70 % mass fraction), which is used as digestion solvent of graphene powder, to make up the control test sample, and blank solution for instrument self-checking and cleaning.

4.1.2 Hydrofluoric acid (HF, MOS or higher, 40% mass fraction), when necessary (e.g. Si or Ti element is a little higher in content), as a digestion solution together with nitric acid. However, HF is powerfully corrosive, and working with HF requires special precautions to avoid contact with skin. All operation should be carried out in fume hood with safe protection such as rubber gloves and work clothes, etc.

4.1.3 Ultra-pure water used as diluted solvent.

4.2 Description of measurement instrument and apparatus

4.2.1 Measurement instrument

ICP-MS should be used to measure metal impurities in graphene powder. ICP-MS instrument can be equipped with a quadrupole or sector field mass spectrometer, or another type of ICP-MS instrument operating with at least 1u (atomic mass unit) resolution for multi-elements determination.

4.2.2 Sample pre-treatment apparatus

4.2.2.1 Microwave digester used for digestion of graphene powder immersed in digestive solvent.

4.2.2.2 Pressure tank (acid proof and high temperature resistance) also used for digestion. The tank should be placed in an oven over 200 °C for more than 6 h.

4.2.2.3 Acid-driven processor used for acid-driving of the test samples after digestion.

4.2.3 Other

4.2.3.1 Static discharge gun used to neutralize the static charge while graphene powder is being weighed and transferred.

4.2.3.2 Analytical balance with a resolution of 0,000 1 g.

4.2.3.3 **100 µL, 1 000 µL, 5 mL pipettes** used to transfer liquid.

4.2.3.4 **10 mL, 25 mL, 50 mL volumetric flasks** used for constant volume.

4.2.3.5 **10 mL, 15 mL, 50 mL, 100 mL centrifuge tubes** used as container.

4.3 Calibration standards

4.3.1 Standard stock solutions

There are approximately 20 to 30 kinds of metal impurities in industrial graphene powder, so mixed standard solutions including Fe, Cr, Mn, W, Ti, Mo, Zn, Ni, Rb, Zr, Sr, Sb, Sn, Co, Pb, Ce, Ba, and Cu elements, etc, are recommended.

It is recommended to use simultaneously four kinds of mixed standard solution (available from commercial vendors), such as, but not limited to, the following.

a) Multi-element Solution No. 1: Ag, Al, As, Ba, Be, Bi, Ca, Cd, Co, Cr, Cs, Cu, Fe, Ga, In, K, Li, Mg, Mn, Na, Ni, Pb, Rb, Se, Sr, Tl, U, V, and Zn.
b) Multi-element Solution No. 2: Ce, Dy, Er, Eu, Gd, Ho, La, Lu, Nd, Pr, Sc, Sm, Tb, Th, Tm, Y, and Yb.
c) Multi-element Solution No. 3: Au, Hf, Hg, Ir, Pd, Pt, Rh, Ru, Sb, Sn, and Te.
d) Multi-element Solution No. 4: B, Ge, Mo, Nb, P, Re, S, Si, Ta, Ti, W, and Zr.

4.3.2 Internal standard (IS) solutions

Single-element internal standard (IS) solutions are available from commercial vendors. Alternatively, IS stock solutions can be prepared in-house giving due consideration to the purity of water and acids. Li, Sc, Ge, Y, Rh, In, Tb, Lu, Re, Bi, etc. are usually used as IS elements.

Prior to quantitative analysis, a preliminary scan should be conducted to select suitable IS elements.

The selection of IS elements should fulfil four criteria.

a) The IS element is not present in the test sample.
b) The IS element has similar molecular weight and ionization energy to the analytical element.
c) The IS element is not disturbed easily by other elements.
d) The signal intensity of the IS element is not affected by count statistics, that is, the concentration of the IS element is sufficiently high.

5 Sample preparation method

5.1 General

For the test sample of graphene powder, four to six parallel specimens should be treated simultaneously. According to the content of metal impurities measured, the standard recovery of several key elements should be measured, by adding the standard solution of metal elements selected into test specimens prior to digestion; at least two parallel specimens shall be prepared for spiked specimens.

5.2 Sample pre-treatment procedure

The pre-treatment procedure is as follows.

1) Select several PTFE digestion vessels, according to the microwave digester used and the number of parallel test specimens, spiked samples and control samples.