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TECHNICAL SPECIFICATION

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Nanomanufacturing – Key control characteristics – Part 6-30: Graphene-based material – Anion concentration: Ion chromatography method

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IEC TS 62607-6-30 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
113/824/DTS	113/846/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/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- withdrawn, or
- revised.

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INTRODUCTION

In recent years, graphene-based materials have drawn increasing attention from academia and industry due to their unique physical and chemical properties. Powders consisting of graphene-based material are now mass produced and widely used in fields such as battery, capacitor, coating, heat conducting, etc.

Anions are common and significant non-metallic impurities in graphene-based materials, originating from raw materials or chemicals used during the production process. These anions play a crucial role in influencing the applications of graphene-based materials. For instance, anions can lead to changes in reversible capacity and coulombic efficiency when graphene-based materials are employed in batteries and capacitors. Therefore, anion concentration stands as a key characteristic of graphene-based materials. Fluoride, chloride, nitrite, bromide, nitrate, sulphate, and phosphate are among the prevalent anions detected in numerous graphene-based materials gathered from the market.

Various methods have been utilized for determination of anions. The most common techniques for quantifying anions include titration, colorimetric determination, and ion chromatography (IC). IC offers several advantages – such as unique selectivity, fast analysis speed, high sensitivity, good accuracy, and easy operation – over alternative techniques in the analysis of anions. Moreover, one of its significant advantages is the capability to simultaneously determine multiple types of anions.

Sample preparation is a critical step in the analytical process, particularly when dealing with powders characterized by very low density and strong hydrophobic properties. It is essential to obtain a sample extraction solution to effectively isolate the analytes from the matrix before conducting IC instrumental determinations. Consequently, the accuracy, precision, and quantification limits of the analysis are significantly influenced by the sample preparation process. This document furnishes specific sample preparation details tailored for powders composed of graphene-based materials. Importantly, the described method is not confined solely to graphene-based materials but is also applicable to other carbonaceous materials such as graphite and graphite oxide.

The purpose of this document is to describe a test method to determine contents of anions in graphene-based material. A case study illustrating the application of this document can be found in Annex D.

NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS –

Part 6-30: Graphene-based material – Anion concentration: Ion chromatography method

1 Scope

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

- anion concentration

  for powder of graphene-based material by

- ion chromatography.

In this document, the measured anions are fluoride, chloride, nitrite, bromide, nitrate, sulphate, and phosphate. These anions, present in the extraction solution of graphene-based materials, are separated into distinct elution bands on the ion chromatographic separation column and subsequently measured using a conductivity detector. Quantification of these anions is accomplished by establishing a proportional relationship between the measured signal (peak area or peak height) and the concentration of each anion. This is achieved by calibrating the system using a series of standards containing known amounts of each anion. Subsequently, unknown samples are analysed under the same conditions as the standards to determine their anion concentrations.

– Powder of graphene-based material addressed by this document includes graphene oxide, reduced graphene oxide and functionalized graphene, graphene, bilayer graphene, trilayer graphene and few-layer graphene.

NOTE This document can also be used for other carbonaceous material such as graphite and graphite oxide.

– This document targets graphene-based material manufacturers and downstream users to guide their material design, production and quality control.

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 terminology databases for use in standardization at the following addresses:

- IEC Electropedia: available at https://www.electropedia.org/
- ISO Online browsing platform: available at https://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 layer, 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.

[SOURCE: IEC TS 62607-6-3:2020, 3.2.4]

3.2 Key control characteristics measured in accordance with this document

3.2.1
anion concentration
amount of negatively charged ions divided by the mass of graphene-based material

Note 1 to entry: The term is presumed to mean mass concentration. The unit "milligram per kilogram" is recommended.

Note 2 to entry: Anions (fluoride, chloride, nitrite, bromide, nitrate, sulphate and phosphate) present in graphene-based material can be non-intentionally added substances that come from raw materials or chemicals used during production process or intentionally added substances to improve the performance.

3.3 Terms related to the measurement method

3.3.1
ion chromatography
IC
chromatography technique that separates ions based on their affinity for the immobilized ion exchange sites on the ion exchanger followed by quantification of ions through conductivity measurement

[SOURCE: IEC TR 62697-2:2018, 3.1]

3.3.2
eluent
liquid phase used to achieve separation and transport of analytes

[SOURCE: ISO/TS 21362:2018, 3.12, modified – The Notes to entry have been deleted.

4 General

4.1 Measurement principle

Anions are extracted by distilled or deionized water from the powder consisting of graphene-based material. After filtering the extract with a 0,22 µm polyethersulphone membrane filter, the filtrate is analysed by IC to determine anions. Anions are separated into individual elution bands on the separation column of the ion chromatograph. The conductivity of the eluent is reduced with an anion suppression device prior to the ion chromatograph's conductivity detector, where the anions of interest are measured. Quantification of anions in the powder sample is achieved by calibrating the system with a series of standards containing known amounts of anions and then analysing unknown samples under the same conditions as the standards.

4.2 Description of measurement apparatus

The measurement system consists of the following components:

- Analytical balance, with sensitivity not worse than 0,000 1 g or even finer.
- Ball mill.
- Ultrasonic cleaners, with maximum input power no less than 250 W and working frequency no less than 40 kHz.
- Glass sample vials, with volume of 25 ml and rinsed thoroughly with water before use.
- Polyether sulfone membrane filter, 0,22 µm.
- Ion chromatographic system, as shown in Figure 1.

  Pump: IC systems have a high-pressure pump that delivers the mobile phase (usually an aqueous solution) at a constant flow rate through the system.

  Sample injector: This is where the sample is introduced into the system. It is typically an autosampler that precisely injects a known volume of the sample into the chromatographic column.

  Chromatographic column: This is a critical component where the separation of ions occurs. It is typically packed with a stationary phase that interacts with the ions in the sample. The type of stationary phase can vary depending on the analysis.

  Detector: The eluent (the mobile phase carrying the sample) that exits the chromatographic column flows through a detector. Common detectors in IC include conductivity detectors, UV detectors, and amperometric detectors. The choice of detector depends on the type of ions being analysed.

  Suppressor: In some IC applications, especially for measuring anions, a suppressor is used to convert ions into their corresponding acids, improving sensitivity.