Technical Specification

ISO/TS 23359

# Nanotechnologies — Chemical characterization of graphene-related two-dimensional materials from powders and liquid dispersions

*Nanotechnologies — Caractérisation chimique des matériaux bidimensionnels similaires au graphène à partir de poudres et de dispersions liquides*

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# Foreword

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# Introduction

Graphene nanoplatelets (GNPs) are applied in many technology areas, including solar cells, biosensors, displays, composites, flexible electronics and energy storage, due to the exceptional properties of graphene. However, it is not just GNPs that are used commercially but other material variants as well, such as reduced graphene oxide, graphene oxide and chemically functionalised forms of GNPs. These different graphene-related two-dimensional materials (GR2Ms) are suitable for different application areas and therefore, there must be a full understanding of the chemical properties of commercially available materials, so that the correct material is selected for specific application areas.

As these materials are increasingly used in different industries, international standardization is needed to support commercialization. Reliable, accurate, and reproducible measurements are important due to the multiple production routes and therefore variability in properties. Producers of the material must use standards to maintain quality in manufacture and confidence in the supply chain.

This document specifies methods to measure the chemical properties of powders and dispersions containing a GR2M. The techniques covered are X-ray photoelectron spectroscopy (XPS), thermogravimetric analysis (TGA), inductively coupled plasma mass spectrometry (ICP-MS), and Fourier-transform infrared spectroscopy (FTIR). These techniques determine the elemental composition, oxygen to carbon ratio, trace metal impurities, weight percentage of chemical species and the functional groups present.

XPS is used to provide quantitative measurements of the surface elemental composition of GR2Ms. It can measure every element except hydrogen and helium that are within up to approximately 10 nm of the surface and at equivalent homogeneous concentrations above the XPS detection limit.

TGA is a common material characterization technique available in research and industry labs, which offers rapid and simple characterization of bulk material properties providing useful qualitative and quantitative information. TGA is widely used for characterization of GR2M to determine the amount of impurities (i.e. water, amorphous carbon, metals), presence of functional groups, traces of surfactants or other organic impurities from fabrication processes or impurities from the initial raw material (graphite, silica, metal oxides etc).

ICP-MS is used to provide detection of the trace metal impurities in samples containing graphene related two-dimensional materials. However, using conventional solution sample introduction ICP-MS, the sample must be completely solubilized and hence digestion of the samples is required using harsh acid and microwave treatment before analysis using ICP-MS.

FTIR is used to understand the functional groups that are present for different materials with significant non-carbon elements, already identified using complementary techniques herein.

# Nanotechnologies — Chemical characterization of graphene-related two-dimensional materials from powders and liquid dispersions

## 1 Scope

This document specifies methods for characterizing the chemical properties of powders or liquid dispersions containing graphene-related two-dimensional material (GR2M), using a set of suitable measurement techniques.

This document covers the determination of elemental composition, oxygen to carbon ratio, trace metal impurities, weight percentage of chemical species and functional groups present, by use of the following techniques:

— X-ray photoelectron spectroscopy (XPS);

— thermogravimetric analysis (TGA);

— inductively coupled plasma mass spectrometry (ICP-MS);

—Fourier-transform infrared spectroscopy (FTIR).

This document covers sample preparation, protocols and data analysis for the different techniques.

## 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 15472, *Surface chemical analysis — X-ray photoelectron spectrometers — Calibration of energy scales*

ISO 16129, *Surface chemical analysis — X-ray photoelectron spectroscopy — Procedures for assessing the day-to-day performance of an X-ray photoelectron spectrometer*

ISO 18115-1, *Surface chemical analysis — Vocabulary — Part 1: General terms and terms used in spectroscopy*

ISO 20903, *Surface chemical analysis — Auger electron spectroscopy and X-ray photoelectron spectroscopy — Methods used to determine peak intensities and information required when reporting results*

ISO 21270, *Surface chemical analysis — X-ray photoelectron and Auger electron spectrometers — Linearity of intensity scale*

ISO 24237, *Surface chemical analysis — X-ray photoelectron spectroscopy — Repeatability and constancy of intensity scale*

ISO 80004-1, *Nanotechnologies — Vocabulary — Part 1: Core terms*

ISO/TS 80004-6, *Nanotechnologies — Vocabulary — Part 6: Nano-object characterization*

ISO/TS 80004-13, *Nanotechnologies — Vocabulary — Part 13: Graphene and other two-dimensional (2D) materials*