



BSI Standards Publication

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

National foreword

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The UK participation in its preparation was entrusted to Technical Committee NTI/1, Nanotechnologies.

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partir de poudres et de dispersions liquides (ISO/TS
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European foreword

This document (CEN ISO/TS 23359:2025) has been prepared by Technical Committee ISO/TC 229 "Nanotechnologies" in collaboration with Technical Committee CEN/TC 352 "Nanotechnologies" the secretariat of which is held by AFNOR.

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The text of ISO/TS 23359:2025 has been approved by CEN as CEN ISO/TS 23359:2025 without any modification.

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Foreword

ISO (the International Organization for Standardization) and IEC (the International Electrotechnical Commission) form the specialized system for worldwide standardization. National bodies that are members of ISO or IEC participate in the development of International Standards through technical committees established by the respective organization to deal with particular fields of technical activity. ISO and IEC technical committees collaborate in fields of mutual interest. Other international organizations, governmental and non-governmental, in liaison with ISO and IEC, also take part in the work.

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This document was prepared jointly by Technical Committee ISO/TC 229, *Nanotechnologies*, and Technical Committee IEC/TC 113, *Nanotechnology for electrotechnical products and systems*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 352, *Nanotechnologies*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

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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*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18115-1, ISO 80004-1, ISO/TS 80004-6, ISO/TS 80004-13 and the following apply.

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:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

3.1

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-layered graphene (FLG).

Note 3 to entry: Graphene has edges and can have defects and grain boundaries where the bonding is disrupted.

Note 4 to entry: In situations where the word graphene is used as an adjective, including in terms such as graphene-enabled, the term commonly and incorrectly refers to *GR2M* (3.7) and not just to single-layer graphene.

[SOURCE: ISO/TS 80004-13:2024, 3.1.2.1.]

3.2

graphite

allotropic form of the element carbon, consisting of graphene layers stacked parallel to each other in a three-dimensional, crystalline, long-range order

Note 1 to entry: Adapted from the definition in the IUPAC Compendium of Chemical Terminology.

Note 2 to entry: There are two primary allotropic forms with different stacking arrangements: hexagonal and rhombohedral.

[SOURCE: ISO/TS 80004-13:2024, 3.1.2.2.]

3.3

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 80004-13:2024, 3.1.2.7.]

3.4

few-layer graphene

FLG

two-dimensional material consisting of three to ten well-defined stacked graphene layers

[SOURCE: ISO/TS 80004-13:2024, 3.1.2.11.]

3.5

graphene nanoplatelet GNP

nanoplate consisting of graphene layers

Note 1 to entry: GNPs typically have thickness of between 1 nm to 3 nm and lateral dimensions ranging from approximately 100 nm to 100 µm.

[SOURCE: ISO/TS 80004-13:2024, 3.1.2.12]

3.6

graphene oxide GO

chemically modified graphene with 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 O/C atomic ratios of approximately 0,5 (C/O ratios of approximately 2,0) depending on the method of synthesis.

Note 2 to entry: Graphene oxide is predominately prepared by oxidation and exfoliation of graphite.

Note 3 to entry: Oxidative modification can also occur at the edges.

Note 4 to entry: Restacking of graphene oxide can occur. Therefore, care must be taken when preparing samples or products from highly concentrated liquid dispersions as this can lead to agglomeration and aggregation of the primary particles, which are a single-layer.

[SOURCE: ISO/TS 80004-13:2024, 3.1.2.15]

3.7

graphene related 2D material GR2M

carbon-based two-dimensional material consisting of one to 10 layers, including graphene, graphene oxide, reduced graphene oxide, and functionalized variations thereof

Note 1 to entry: This includes bilayer graphene, trilayer graphene and few-layer graphene.

Note 2 to entry: The terms graphene-based material and graphene-material are deprecated here. They have been used to describe materials other than graphene, such as graphene oxide.

Note 3 to entry: "Graphene-related 2D material" is defined in contrast with graphene-based and GR2M-based .

[SOURCE: ISO/TS 80004-13:2024, 3.1.1.2]

3.8

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 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.

Note 4 to entry: The O/C atomic ratio is approximately 0,1 to 0,5 (C/O ratio 2 to 10).

[SOURCE: ISO/TS 80004-13:2024, 3.1.2.16]

3.9

graphite oxide

chemically modified graphite prepared by extensive oxidative modification of the basal planes

Note 1 to entry: The structure and properties of graphite oxide depend on the degree of oxidation and the particular synthesis method.

Note 2 to entry: In powder form, restacking of graphite oxide layers can occur.

[SOURCE: ISO/TS 80004-13:2024, 3.1.2.14]

3.10

X-ray photoelectron spectroscopy

XPS

method in which an electron spectrometer is used to measure the energy distribution of photoelectrons and Auger electrons emitted from a surface irradiated by X-ray photons

Note 1 to entry: X-ray sources in common use are unmonochromated Al K α and Mg K α X-rays at 1 486,6 eV and 1 253,6 eV, respectively. Modern instruments also use monochromated Al K α X-rays. Some instruments make use of various X-ray sources with other anodes or of synchrotron radiation.

Note 2 to entry: Extended pressure XPS (EP-XPS), near ambient pressure XPS (NAP-XPS) and hard X-ray photo electron spectroscopy (HAXPES) are variants of XPS.

[SOURCE: ISO 18115-1:2023, 11.6, modified — Abbreviations in Note 2 to entry have been spelt out]

3.11

thermogravimetric analysis

TGA

method in which the change in the mass of a sample is measured as a function of temperature while the sample is subjected to a controlled temperature programme

[SOURCE: ISO/TS 80004-6:2021, 6.1.2, modified — Term has been changed to “thermogravimetric analysis”.]

3.12

inductively coupled plasma mass spectrometry

ICP-MS

analytical technique comprising a sample introduction system, an inductively coupled plasma source for ionization of the analytes, a plasma or vacuum interface and a mass spectrometer comprising an ion focusing, separation and detection system

[SOURCE: ISO/TS 80004-6:2021, 5.23]

3.13

Fourier transform infrared spectroscopy

FTIR

spectroscopy in which a sample is subjected to excitation of molecular bonds by pulsed, broad-band infrared radiation, and the Fourier transform mathematical method is used to obtain an absorption spectrum

[SOURCE: ISO/TS 80004-6:2021, 5.8]

4 Abbreviated terms

For the purposes of this document, the following symbols and abbreviations apply.

1D	one dimensional
1LG	single layer graphene
2D	two-dimensional
2LG	bilayer graphene
3D	three dimensional
DTG	first derivative of the TG curve
d2TG	second derivative of the TG curve
FLG	few-layer graphene
FTIR	Fourier-transform infrared spectroscopy
GNP	graphene nanoplatelet
GR2M	graphene related two-dimensional material
HAXPES	hard X-ray photo electron spectroscopy
ICP-MS	inductively coupled plasma mass spectrometry
SCA	surface chemical analysis
TG	thermogravimetric
TGA	thermogravimetric analysis
XPS	X-ray photoelectron spectroscopy

5 Approaches to chemical characterization

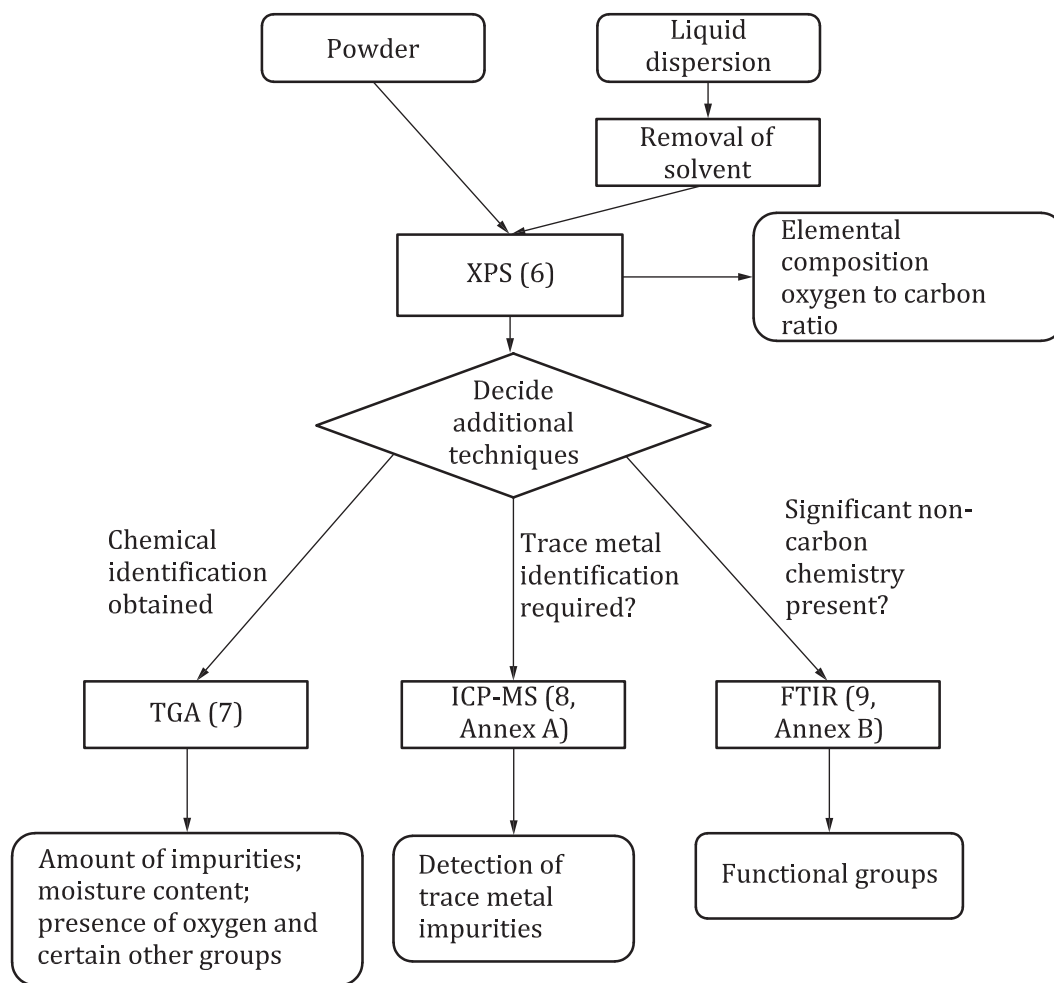
There are a number of different techniques that can be used to measure the chemical properties of GR2Ms. Each technique provides different measurands and information. The techniques outlined in this document are XPS, TGA, ICP-MS, and FTIR. [Table 1](#) provides a summary of the methods and approaches to chemical characterization.

Table 1 — Approaches to chemical characterization

Technique	Measurands	Analysis size	Limitations
XPS (see Clause 6)	Quantitative elemental analysis (atomic per-cent). Oxygen-to-carbon ratio.	Surface measurement with analysis depth down to 10 nm. Lateral resolution: ≈ 1 to $\approx 1\,000\ \mu\text{m}$.	Limits of detection between 0,01 % and 0,5 % for every element present in the surface analysis region (except H and He).
TGA (see Clause 7)	Moisture content. Mass percentage of combustible chemical species identified. Non-combustible impurities.	Bulk measurement	Requires complementary methods for identification of chemical species.
ICP-MS (see Clause 8 and Annex A)	Trace metal impurities.	Bulk measurement	Microwave and acidic digestion of material required before analysis.
FTIR (see Clause 9 and Annex B)	Chemical functional groups.	Bulk measurement	Sensitivity requires significant levels (several atomic percent) of chemical functionalization. Requires samples to be thoroughly dehydrated.
NOTE: XPS can also provide: a) chemical state information (not covered in this document); b) X to carbon ratios where X is any element measured other than carbon. Oxygen to carbon ratios are particularly useful for information when analysing rGO and GO.			

Follow the sequence of measurement methods and process outlined in [Figure 1](#) to determine the chemical properties of GR2Ms. Firstly, undertake XPS to determine quantitative elemental analysis, the main measurand for chemical measurement, and, if required, chemical state information. Then, decide if any additional measurands are required. If so, undertake a combination of TGA, ICP-MS, or FTIR, as appropriate.

The sample may be a powder or a liquid dispersion (suspension). For liquid dispersions, samples are dried to a powder form, in some cases on a substrate, to undergo further characterization. Liquid dispersions can include other formulated ingredients which, when dried, will lead to the inclusion of these chemicals in the sample to be analysed.



NOTE The numbers relate to the clauses where the method is detailed.

Figure 1 — Sequence of methods used to determine chemical properties of GR2Ms from powder or liquid dispersion

The storage and laboratory humidity levels should be monitored as, depending on the sample, these can play a role in varying the chemical functionalization of GR2M.

As well as chemical properties, the structural properties of the GR2M should be considered and can be important for cross comparison with the chemical measurements. Refer to ISO/TS 21356-1 for information on structural characterization of graphene from powders and liquid dispersions, using techniques such as Raman spectroscopy, the Brunauer-Emmett-Teller (BET) method, atomic force microscopy, scanning electron microscopy (SEM), transmission electron microscopy (TEM) and selected area electron diffraction (SAED).

NOTE ISO/TR 19733 outlines other techniques that can provide chemical analysis. This includes X-ray diffraction and combustion techniques. These techniques currently have unknown uncertainties but can provide useful information for quality control purposes once a material has been measured using the methods outlined in this document.

6 X-ray photoelectron spectroscopy (XPS)

6.1 Introduction

XPS can be used to investigate the surface chemistry of GR2M samples. It provides chemical information from the top ≤ 10 nm of a sample with a spatial resolution typically between approximately ten to a few hundred micrometres. The following information should be measured:

- elemental composition with typically 0,1 atomic percent sensitivity;
- chemical binding state information (if required).

In addition, XPS is able to provide information on:

- a) thickness measurement of overlayers of less than 10 nm on a substrate;
- b) surface chemical imaging with a resolution of typically approximately 10 μm ;
- c) depth-distribution of chemical species through angle-resolved analysis, background shape analysis and sputter depth profiling.

XPS works by irradiating a sample with X-rays (commonly Al $K\alpha$ or Mg $K\alpha$) in vacuum. When an X-ray photon hits and transfers this energy to a core-level electron, the photoelectron is emitted from its initial state with a kinetic energy dependent on the incident X-ray and binding energy of the atomic orbital from which it originated. The energy and intensity of the emitted photoelectrons are analysed to identify and determine the concentrations of the elements present. The chemical state of the atom influences the binding energy scale position and the shape of photoelectron peak in the spectrum and these can be used to infer the chemical environment of the atom by expert analysis.

6.2 Instrument preparation

The user should be competent in XPS analysis and should, as far as possible, conform to ISO 10810. Prior to measurements, the XPS instrument shall be in good working order. The linear range of operation shall be determined using ISO 21270. The energy scale of the instrument shall be calibrated using ISO 15472. The intensity scale of the operating modes used for quantitative elemental analysis shall be calibrated according to laboratory practice. It should be calibrated by the instrument manufacturer or appropriate metrology institute. It is normal for these steps to be carried out after the installation or at a regular instrument service, in which case the stability of the instrument shall be assessed using ISO 16129 and ISO 24237. Details of the instrument calibration and sensitivity factors employed should be recorded in accordance with ISO 15470.

6.3 Sample preparation

The test sample consisting of a GR2M should be measured after directly pressing a powder into a pellet, which produces a flat homogenous surface for XPS and addresses any possible health and safety concerns due to the possible nano-particulate nature of the material. An example of a pressed pellet mounted on a 1 cm $\times$ 1 cm silicon wafer is shown in [Figure 2](#). The use of a pressed pellet will produce the most repeatable results. As an alternative to pressing pellets, the powders may be mounted and analysed as received but the results are likely to be less repeatable. The powders may also be mounted in a recessed well, but the powder must be well fixed in the recess to avoid any damage to or contamination of the XPS equipment. Whichever sample preparation is used, the sample preparation procedure should be recorded. In most cases, the sample preparation procedure will affect the measured result. Results measured on pellets should not be compared with results from powders.

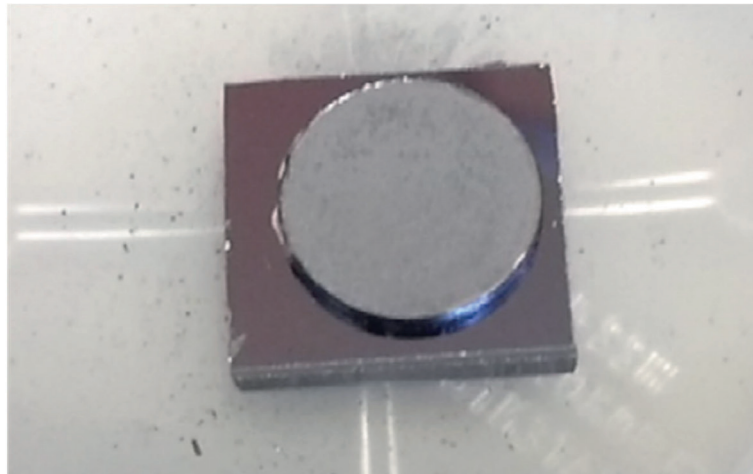


Figure 2 — Example of a pressed pellet mounted on a silicon wafer

The contamination of samples must be avoided during sample preparation. Disposable laboratory gloves and masks should be used. The surfaces of pellet dies should be free from contaminants. Sample preparation should be performed in a well-ventilated area. A relative humidity of higher than 60 % should be avoided. Samples should only be handled with clean metal implements. The area to be analysed should not be touched. For further information on sample preparation, refer to ISO 20579-1:2024, Clause 5 and Annex B.

The sample preparation should be documented as described in ISO 20579-4, Clause 5 and Annex B.

Once the test samples are prepared, position the sample in the instrument in accordance with standard operating procedures.

Other types of samples should not be put onto the sample holder with the test samples, in order to avoid contamination.

6.4 Method

The method shall be as follows:

- a) Achieve a suitable vacuum, according to standard operating procedures. The instrumental vacuum should be better than approximately 10^{-6} Pa (10^{-8} mbar) during data acquisition.
- b) A survey scan with a minimum pass energy of 80 eV or higher shall be acquired. The kinetic energy range shall be from 200 eV to at least 10 eV greater than the X-ray energy. The energy step size shall be 1 eV or smaller. The dwell time or number of sweeps shall be adjusted to ensure that the number of counts of C 1s maximum is greater than 1×10^5 counts per second (or counts/s).
- c) Acquire high resolution (narrow) scans with a pass energy of 40 eV or lower for appropriate peaks. The energy step size should be between 0,05 eV and 0,1 eV. High resolution scans of C 1s, O 1s, N 1s, Cl 2p, and S2p regions are recommended as a minimum, if the elements are identified as present as well as regions for other elements expected or suspected to be present and elements identified in the survey scan.

For example, if fluorine is expected to be present either as the GR2M has been functionalized with fluorine or fluorine is expected to be a contaminant, then a high-resolution scan of F 1s is recommended.

In order to obtain good signal quality, the test samples should be scanned until a peak intensity of greater than 2×10^4 counts/s is recorded at the C 1s maximum. Peaks of all other elements should be scanned for the same number of scans as the C 1s peak, or a greater number of scans should be performed. For each high-resolution spectra, a total energy window of at least three times the width of the main peak should be used. In addition, for the C 1s, the scan energy window should extend at least 15 eV to the higher binding energy side of the peak to account for peak asymmetry. An example of the survey scan for a GR2M sample and a narrow scan of the C1s peak is shown in [Figure 3](#).