EMPIR Call 2019 – Energy, Environment, Normative and Research Potential

Selected Research Topic number: **SRT-n10**
Version: 1.0

Title: Standardisation of structural and chemical properties of graphene

Abstract

Graphene and related 2D materials are predicted to make a major impact in many different technology areas, either through incremental advances via the replacement of current materials, or via disruptive changes to entirely new or current fields. However, the uptake of graphene and related 2D materials into commercial products is currently being hindered as industrially-produced graphene is often not correctly characterised and thus may not be suitable for use in specific applications and products. Research is required to develop and progress documentary standardisation of structural and chemical characterisation of graphene via pre-normative VAMAS interlaboratory testing leading to published ISO standards.

Keywords

Two-dimensional materials, structural, chemical, characterisation, graphene, graphene oxide, standardisation, nanotechnology, ISO, VAMAS

AFM (Atomic Force Microscopy), SEM (Scanning Electron Microscope), TEM (Transmission Electron Spectroscopy), XPS (X-Ray Photoelectron Spectroscopy)

Background to the Metrological Challenges

Graphene is defined by ISO and CEN as a single-layer of carbon atoms. It is a key advanced material, existing in flake form in powders or liquid dispersions and in larger grown sheets. The flake form in particular is already starting to find commercial application by small-to-medium enterprises (SMEs) to multinational corporations, for a large range of application areas such as membranes, composites, sensors, transistors, Li-ion batteries, transparent conductive films and supercapacitors. For example, Ford have recently incorporated graphene flakes in parts for engine noise reduction where graphene leads to stronger, heat resistant, more durable products. Graphene has the potential for many more applications in other areas in the future including medical and healthcare applications using functionalised graphene. Publications and patents continue to increase, and the future world-wide graphene market is estimated to be worth ~€400 million in 2024, which equals a growth of more than 300 % each year.

There are currently over 100 commercial ‘graphene’ producers worldwide, including leading graphene producers in Europe, with an 'on paper' offering of materials with vastly different properties and types. However, many suppliers (and buyers) are hindered due to uncharacterised material that can be more often graphite rather than graphene or have batch to batch variations. Products and applications suffer. This is acknowledged as the single biggest issue by graphene companies, suppliers and standards bodies (for example ISO TC229 (nanotechnologies), BSI NTI/1 (nanotechnologies) and BSI UK-China JWG on graphene standardisation). Issues include structural determination – is the material graphene or graphite, how many layers are present and what are the flake sizes in a batch? In chemical determination issues include the amount of oxidation (for graphene oxide and reduced graphene oxide), impurities and functionalisation. There are no standard ways to measure these properties and others which will be paramount to industry who wish to take the material from the laboratory to large-scale production. Once the material itself is characterised reproducibly and in a way that allows cross-comparison, products can be tailored to specific types of graphene and related 2D materials.

Documentary measurement standards at European (CEN) and international (ISO) level do not currently exist and are much needed to instil commercial confidence and enable the growth in this emerging industry. The first ISO standard on graphene was published in 2017 on terminology: ISO/TS 80004-13 'Nanotechnologies - Vocabulary - Part 13: Graphene and other two-dimensional materials. This defined graphene as a single carbon layer and few-layer graphene between 3 to 10 layers. European graphene companies are keen for their products to adhere to this standard but require measurement standards to be developed to do this.

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Graphene flakes are specifically defined as a nanomaterial (2011/696/EU) and graphene manufacturers and supplier must comply with the chemical's regulation REACH (2006/1907) and specifically the amended annexes (2018/1881) specifically related to nanomaterials. Here, validation and standardisation of measurement methods will be critical for industry regulation.

Objectives

Proposers should address the objectives stated below, which are based on the PRT submissions. Proposers may identify amendments to the objectives or choose to address a subset of them in order to maximise the overall impact, or address budgetary or scientific / technical constraints, but the reasons for this should be clearly stated in the protocol.

The JRP shall focus on the validation and standardisation of measurement and characterisation methods for chemical and structural characterisation of graphene in powders and liquid dispersions for industrial applications. This will be achieved via validation in pre-normative VAMAS interlaboratory studies and development and publication of international graphene measurement standards under the auspices of ISO TC 229 "Nanotechnologies".

The specific objectives are

1. To lead and provide a contribution to the publication "ISO TS 21356-1 Nanotechnologies – Structural characterisation of graphene: Part 1: Graphene from powders and dispersions". The contribution will focus on measurement methods for the characteristics of graphene flakes in order to verify measurements made on commercial graphene flake products.
2. To lead and provide a contribution to the development of "ISO/PWI 23359 Nanotechnologies - Chemical characterisation for graphene in powders and suspensions and a contribution to ISO/PWI Nanotechnologies – Structural characterisation of graphene oxide flakes: thickness and lateral size measurement using AFM and SEM". The contribution will focus on developing validated quantitative measurement methods.
3. To provide a contribution to pre-normative international interlaboratory studies in VAMAS TWA 41 (graphene and related 2D materials), leading and participating in characterisation studies focused on structural and chemical properties using, for example, XPS, AFM, Raman, SEM and TEM techniques.
4. To work closely with the European and International Standards Developing Organisations, and pre-normative organisations, and the users of the Standards they develop, including the Graphene Flagship, to ensure that the outputs of the project are aligned with their needs and incorporated into Standards at the very earliest opportunity.

The proposed research shall be justified by clear reference to the measurement needs within strategic documents published by the relevant Regulatory body or Standards Developing Organisation or by a letter signed by the convenor of the respective TC/WG. EURAMET encourages proposals that include representatives from industry, regulators and standardisation bodies actively participating in the projects. The proposal must name a "Chief Stakeholder", not a member of the consortium, but a representative of the user community that will benefit from the proposed work. The "Chief Stakeholder" should write a letter of support explaining how their organisation will make use of the outcomes from the research, be consulted regularly by the consortium during the project to ensure that the planned outcomes are still relevant and be prepared to report to EURAMET on the benefits they have gained from the project.

Proposers should establish the current state of the art and explain how their proposed research goes beyond this.

EURAMET expects the average EU Contribution for the selected JRPs in this TP to be 0.8 M€ and has defined an upper limit of 1.0 M€ for this project.

EURAMET also expects the EU Contribution to the external funded partners to not exceed 30 % of the total EU Contribution across all selected projects in this TP.

Any industrial partners that will receive significant benefit from the results of the proposed project are expected to be unfunded partners.

Potential Impact

Proposals must demonstrate adequate and appropriate participation/links to the "end user" community, describing how the project partners will engage with relevant communities during the project to facilitate

knowledge transfer and accelerate the uptake of project outputs. Evidence of support from the “end user” community (e.g. letters of support) is also encouraged.

You should detail how your JRP results are going to:

- Address the SRT objectives and deliver solutions to the documented needs,
- Feed into the development of urgent documentary standards through appropriate standards bodies,
- Transfer knowledge to the graphene manufacture and supply sector.

You should detail other impacts of your proposed JRP as specified in the document “Guide 4: Writing Joint Research Projects (JRPs)”

You should also detail how your approach to realising the objectives will further the aim of EMPIR to develop a coherent approach at the European level in the field of metrology and include the best available contributions from across the metrology community. Specifically, the opportunities for:

- improvement of the efficiency of use of available resources to better meet metrological needs and to assure the traceability of national standards
- the metrology capacity of EURAMET Member States whose metrology programmes are at an early stage of development to be increased
- organisations other than NMIs and DIs to be involved in the work.

Time-scale

The project should be of up to 3 years duration.