Potential European Partnership on Metrology
Call 2021 – Green Deal and Normative

Selected Research Topic number: **SRT-v10**
Version: 1.1

Important information about these documents

This call is being held ahead of any agreement from the Commission that the relevant funding will be available. At present the relevant legislation is still under discussion in both Council and Parliament, and there is no certainty on the detailed arrangements for funding selected projects. The funding of any selected project, and the terms and conditions of participation in the projects, are dependent on completion of the legislative process and the subsequent contractual processes between the European Commission and EURAMET. Proposers submit to this call at their own risk.

Background

Last year, EURAMET submitted a draft proposal to the EC for a further research programme to be established under article 185 of the Treaty on the Functioning of the European Union (TFEU) to follow on from EMRP and EMPIR. This was published by the EC at https://ec.europa.eu/info/research-and-innovation/funding/funding-opportunities/funding-programmes-and-open-calls/horizon-europe/european-partnerships-horizon-europe/candidates-digital-industry-and-space_en

The initiative would be called the European Partnership on Metrology and would aim to create, by 2030, a sustainable and effective system for metrology at European level that ensures Europe has a world-class metrology system that:

- Provides metrology solutions, fundamental metrological reference data and methods, offering fit-for-purpose solutions supporting and stimulating European innovation and responding to societal challenges.
- Supports and enables effective design and implementation of regulation and standards that underpin public policies that address societal challenges.

The Commission commissioned an impact assessment into this proposal and 11 others in similar priority areas, and, based on those findings, published their own proposal for the Partnership, their response to the impact assessment and a draft of the Decision on 23rd February 2021. See:

https://eur-lex.europa.eu/legal-content/EN/TXT/?uri=COM:2021:89:FIN

https://ec.europa.eu/commission/presscorner/detail/en/ip_21_702

https://eur-lex.europa.eu/legal-content/EN/TXT/?uri=CELEX%3A52021SC0035&qid=1614677899327

That draft Decision is currently under discussion in the European Council and the European Parliament.

Under the assumption that the Council and Parliament pass the basic act which would form the legal basis for this research programme, and that the participating countries named in the Draft Decision submit the required commitment letters, EURAMET is publishing these potential Selected Research Topics and draft guidance notes. These documents are not approved by the Commission nor will they lead to a binding decision by EURAMET e.V. for any further negotiation or funding. All published guides and templates are subject to amendment by the EC and EURAMET e.V. as further information becomes known.

Title: In operando metrology for energy storage materials

Abstract

The EU Green Deal is supported by the EU research initiative BATTERY 2030+. EU battery and automotive industries face strong competition for high capacity energy storage technologies for use in electric vehicles, portable devices and grid stabilisation. Despite recent advances in battery performance, capacities and lifetimes are still too poor for many key applications. To accelerate innovation by materials and device manufacturers, new metrology is urgently required. This SRT calls for the development of *in operando* techniques, supported by standardised *ex-situ* analysis and electrochemical measurements, to enable beyond state-of-the-art materials characterisation.

Keywords

In operando metrology, vibrational spectroscopy, x-ray spectroscopy, impedance spectroscopy, mass spectrometry, energy storage materials, energy efficiency, Lithium (Li) batteries, solid-state batteries

Background to the Metrological Challenges

The EU Green Deal is supported by the large-scale cross-sectoral EU research initiative BATTERY 2030+ [1] bringing together the most important stakeholders in battery R&D. The BATTERY 2030+ roadmap proposes research actions to radically transform the way we discover, develop, and design ultra-high-performance, durable, safe, sustainable, and affordable batteries. The roadmap calls for new metrology 'including data recorded under working conditions (i.e. *in operando* measurements), and spanning the full range from optimised laboratory-based to large-scale research facility-based measurements and high-throughput synthesis and laboratory testing'.

The lithium ion battery (LIB) represents the current state-of-the-art energy storage technology for many applications. However, intrinsic limitations in terms of practical energy densities and life cycle, accompanied by rising safety concerns and drawbacks associated with cost and sustainability, highlight the need for improvement. While alternative chemistries exist, these technologies are less mature. Examples include lithium-metal, lithium-sulphur and sodium-ion batteries, as well as novel all solid-state batteries (ASSB) which offer the advantages of reduced flammability and toxicity. Despite advances in materials science, these systems suffer from degradation effects leading to capacity fading over time. Electrochemical methods such as galvanostatic cycling allow such effects to be monitored, but novel analytical tools are needed to understand the underlying chemical and physical processes which are responsible for capacity reduction and failure.

The development of advanced *in operando* techniques for characterising high capacity energy storage materials first requires reliable *ex-situ* methods to be established for detailed physical and chemical analysis both at the beginning of life, and after a varying number and extent of electrochemical cycling. *In operando* measurements are typically highly complex, so a robust metrological framework must be in place to allow validation against *ex-situ* measurements performed under more controlled conditions. However, many ex-situ methods (e.g. for mass deposition and chemical quantitation) have not been optimised or standardised for this application. X-ray spectrometry (XRS) can provide physically traceable measurements, including quantitative measurements of elemental mass depositions and related chemical binding and coordination states at surfaces and in bulk. XRS of battery materials is primarily conducted *ex-situ*, however, recent developments have shown the potential for *in operando* analysis.

A wide range of electrochemical and physical changes occur during battery charging and discharging, and materials properties are highly sensitive to the environmental conditions, making functional characterisation by *ex-situ* analysis highly challenging. Therefore, *in operando* techniques are required to allow characterisation *during* use. Current *in operando* spectroscopy methods are immature and lack the reliability to confidently correlate materials property changes to performance. Little attention has been paid to: (i) whether the *in operando* cells are representative of a real operating device; (ii) the effects of optical illumination or beam damage; (iii) the influence of measurement parameters on the fidelity of the spectroscopic data; and (iv) sample-to-sample variability.

Combining *in operando* spectroscopy with electrochemical techniques is a powerful approach to determine the impact of changes in the physical and chemical properties of battery materials on device performance. In particular, electrochemical impedance spectroscopy (EIS) can provide a wealth of information over a range of timescales, however, it suffers the drawback of being notoriously difficult to interpret and to confidently correlate to physical and chemical phenomena. Improved measurement protocols are needed based on galvanostatic conditions and combining advanced electrochemical approaches with *operando* methods, in order to establish an innovative framework for electrode materials discovery and optimisation. Finally, simultaneous measurement of multiple physical and chemical properties during battery operation is key to establishing strong materials-performance relationships.

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 development of *in operando* techniques, supported by standardised *ex-situ* analysis and electrochemical measurements, to enable beyond state-of-the-art materials characterisation for high capacity energy storage technologies.

The specific objectives are

1. To develop traceable chemical, physical and structural analysis methods for *ex-situ* characterisation of high capacity energy storage materials and components with a focus on x-ray spectroscopic techniques, and including the fabrication and qualification of calibration samples and verification by interlaboratory studies.
2. To establish Best Practice Guides for current and emerging *in operando* spectroscopy methods including x-ray spectroscopy and vibrational spectroscopy, validated by *ex-situ* analysis and round robin activities, in order to improve experimental repeatability and accuracy. To understand the influence of cell geometry, electrode configuration, and measurement parameters on observable phenomena, and to assess the extent and influence of vacuum ultra-violet (VUV) or X-ray radiation damage.
3. To develop novel dynamic electrochemical approaches combined with *in operando* spectroscopy and dimensional metrology for the correlative assessment of the relationships between material structure and cell performances.
4. Based on the results of Objectives 1-3, to develop novel *in operando* instrumentation and hybrid methodologies for multi-parameter spectro-electrochemical characterisation of high capacity energy storage materials and components. To investigate the causal relationship between electronic/molecular- and microstructure information and charge carrier dynamics as measured with electroanalytical methods, to gain information on the state-of-health and state-of-charge.
5. To facilitate – in cooperation with the EMPIR 20NET01 Clean Energy – the take up of the data and measurement infrastructure developed in the project by the measurement supply chain (NMIs, DIs, calibration laboratories), standards developing organisations (e.g. ISO/TC 201) and key end users (materials suppliers and battery manufacturers). To promote technology transfer of the project outputs as lab-based alternatives to synchrotron radiation-based methods, towards industry and manufacturers.

These objectives will require large-scale approaches that are beyond the capabilities of single National Metrology Institutes and Designated Institutes. To enhance the impact of the research, the involvement of the appropriate user community such as industry, standardisation and regulatory bodies is strongly recommended, both prior to and during methodology development.

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

EURAMET expects the average EU Contribution for the selected JRPs in this TP to be 2.2 M€, and has defined an upper limit of 2.7 M€ for this project.

EURAMET also expects the EU Contribution to the external funded partners to not exceed 35 % of the total EU Contribution across all selected projects in this TP.

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 materials developers and device manufacturing 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 the potential European Partnership on Metrology 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.

Additional information

The references were provided by PRT submitters; proposers should therefore establish the relevance of any references.

[1] BATTERY 2030+ roadmap 2018, https://battery2030.eu/research/roadmap/