

Title: Hybrid metrology for sustainable and low-carbon footprint battery materials

Abstract

The European battery industry is under increasing pressure to improve the economic and environmental sustainability of the manufacturing supply chain. Existing high-capacity energy storage technologies such as Li-ion batteries rely on critical elements with low earth abundance, are produced via energy intensive and environmentally unfriendly processes and are difficult to recycle. New metrology is urgently required to support the transition to more sustainable battery chemistries and low carbon-footprint manufacturing processes. This proposal aims for the development of hybrid analytical, structural, and electrochemical techniques, supported by standardised ex- and in-situ characterisation, to enable next generation battery materials innovation.

Keywords

Battery materials, low-carbon, hybrid metrology, in-situ, ex-situ, operando spectroscopy, surface analysis, energy storage, next generation batteries, circular economy

Background to the Metrological Challenges

A global shortage of battery raw materials is expected due to rising market demand in combination with limited EU domestic supply of battery-related commodities. Moreover, a materials metrology framework exists in the context of benchmark lithium-ion battery (LIB) technologies, but the emergence of new chemistries and processing routes designed to address above challenges introduces additional complexity owing to the wider diversity of elemental combinations, structures, and interactions. Hence, there is a need to ensure that existing best practice can be reliably extended to next generation battery materials systems. This is essential to underpin hybrid methodologies designed to uncover structure-chemistry-performance correlations. Furthermore, the existing metrological framework lacks the inclusion of thermal properties of battery materials, which is needed to understand the effects of heat dissipation on cell cycling.

The end-of-life recycling of battery materials can provide a mid-term EU-based source of critical elements to de-risk the materials supply chain. However, the recovery, refining and processing of these recycled materials can strongly impact their purity and the nature and location of contaminants. Hence, there is an urgent need for traceable analytical techniques and calibration materials to reliably quantify elements in recycled materials. This includes trace and minor matrix elements originating from current collectors and electrolytes (e.g. Al, Cu, P, F), as well as other residual contaminants associated with the battery casing and chemical processing (e.g. Fe, S, H-containing compounds). Furthermore, the influence of such impurities on the performance of cells derived from these recycled materials is largely unknown. Hence, there is a need to evaluate the impact on these contaminants on the functional properties of battery materials from recycled commodities, with a view to informing thresholds for quality control purposes. However, in all cases development of procedures for traceable quantification is challenging and method validation for emerging battery chemistries is in many cases missing.

Understanding how the properties of battery materials govern electrochemical behaviour in emerging technologies is essential. However, battery architectures comprise complex, multiscale, composites which undergo dramatic changes during charge-discharge cycling, making these properties highly challenging to measure reliably. Operando techniques enable characterization under dynamic conditions, which is essential due to the unstable chemical nature of electrochemical interfaces in batteries, but improved confidence in these measurements is required. Hence, there is a need to develop instrumentation for hybrid metrology to

enable multiple independent and simultaneous measurement of singular quantities in order to minimize uncertainty. Moreover, the relationships between associated properties and their link to performance are often unknown, so there is also a requirement to measure different properties simultaneously. The ability to specifically characterize electrode-electrolyte interfaces is key, with emphasis on measuring the stability of the cathode electrolyte interphase (CEI). Several operando techniques have been developed including optical and X-ray spectroscopy/diffraction but their hybrid combination has been limited and integration of additional measurements such as calorimetry to evaluate internal resistances, has yet to be explored.

The multiparameter and interactive nature of battery materials properties coupled with the volume and complexity of data generated by operando and hybrid characterization methods, underlines the critical need for integration of sophisticated modelling and computational tools within the metrology infrastructure. The employment of machine learning algorithms and artificial intelligence is essential to improving experimental design through predictive analytics, enhance data interpretation accuracy, and facilitate the elucidation of complex causative relationships between materials properties and electrochemical performance.

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 proposal shall focus on the development of complementary metrology capabilities on analytical, spectroscopic, and other materials characterisation techniques to support development of new sustainable, low-carbon battery technologies, and recycling of existing battery materials.

The specific objectives are:

1. To develop a hybrid metrology approach comprising analytical methods to measure relevant (electro)chemical, structural and thermal properties of sustainable and low-carbon footprint battery materials. This includes building on the existing metrological framework based on well-established benchmark materials for lithium-ion batteries (LIBs). In addition, ensuring the robust extension of this measurement capability to encompass emerging battery chemistries produced using more abundant raw materials and environmentally friendly and low-carbon footprint processes.
2. To develop new metrology approaches and procedures to support recycling of battery materials. This includes establishment of traceable analytical methods, and sampling methods for calibration purposes for absolute quantification of trace or minor matrix elements (e.g. Al, Cu, P, F, Fe, S) in end-of-life battery materials. In addition, investigating how these elements impact cell performance in view of their spatial distribution (surface and bulk) within the cell components, in order to inform acceptable thresholds for battery recycling and manufacture.
3. To develop a traceable methodologies and new instrumentation that would enable hybrid and operando characterization of emerging sustainable battery materials to allow complementary but independent measurement of specific measurands (e.g. localized elemental composition and speciation, coordination environment, crystalline structure and lattice parameters, temperature) to both reduce the associated uncertainties. In addition, this includes enabling the identification of correlations between interconnected material functionalities and the underlying chemical and physical properties.
4. To develop and enhance modelling and computational tools enabled by hybrid metrology (such as orthogonal, interface-sensitive, localised and global, analytical and electrochemical methods) to support new sustainable battery materials innovation. This includes identification of measurable indicators of electrode/electrolyte interface degradation and monitoring performance deterioration metrics in real-time under dynamic operating conditions to elucidate crucial capacity fade mechanisms.
5. To facilitate the take up of the technology and measurement infrastructure developed in the project by the measurement supply chain standards developing organisations (ISO TC201 (surface characterisation), SC10 (X-ray methods), SC7 (electron spectroscopies), WG5 (optical interface analysis), SG2 (energy materials), ISO TC 345 (specialty materials & minerals), as well as BIPM CCQM (TG on LIBs) and VAMAS TWA 2 (Surface Chemical Analysis)), and end users (e.g. automotive and battery manufacturer).

These objectives will require large-scale approaches that are beyond the capabilities of single National Metrology Institutes and Designated Institutes. Proposers shall give priority to work that meets documented

needs, in particular those supporting the European Green Deal. 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. In particular, proposers should outline the achievements of the EMPIR or Metrology Partnership project(s) 21GRD01, 17IND10 and 20IND04 and how their proposal will build on those.

EURAMET expects the average EU Contribution for the selected JRPs in this TP to be 2.8 M€ and has defined an upper limit of 3.5 M€ for this proposal.

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

Proposers should note that the programme funds the activity of researchers to develop the capability, not the required infrastructure and capital equipment, which must be provided from other sources.

Any industrial beneficiaries that will receive significant benefit from the results of the proposed project are expected to be beneficiaries without receiving funding or associated 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 proposal's 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,
- Facilitate improved industrial capability, or improved quality of life for European citizens in terms of personal health, protection of the environment and the climate, or energy security,
- Transfer knowledge to the battery 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 Metrology Partnership 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.

Timescale

The project should be of up to 3 years duration.

Additional information

The links provided in this section are only correct at the time of publication up until the end of the Call year.

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

[1] *Regulation 2023/1542*,
<https://eur-lex.europa.eu/eli/reg/2023/1542/oj>

[2] *European directive 2006/66/EC*,
<https://eur-lex.europa.eu/legal-content/EN/TXT/?uri=celex%3A32006L0066>

[3] <https://doi.org/10.2865/862094>

[4] https://single-market-economy.ec.europa.eu/publications/european-critical-raw-materials-act_en

[5] <https://doi.org/10.1016/j.cirp.2021.04.038>

- [6] *Recycling of Lithium-Ion Batteries—Current State of the Art, Circular Economy, and Next Generation Recycling*,
<https://doi.org/10.1002/aenm.202102917>
- [7] <https://pubs.rsc.org/en/content/articlelanding/2023/sc/d2sc04397a>
- [8] <https://www.big-map.eu/big-map> ; <https://emmc.eu/> ; <https://www.the-marketplace-project.eu>
- [9] *European Technology Platform Battery 2030+, updated roadmap document, August 2023*
- [10] *"ANNEX 2 – Strategic Action Plan on Batteries, EUROPE ON THE MOVE Sustainable Mobility for Europe: safe, connected and clean", European Commission, May 2018*