Versailles Project on Advanced Materials and Standards

VAMAS

Calls for Participation – July 2025

VAMAS

Energy Storage Systems

Incubator Project - Technical Working Area 0

CALL FOR PARTICIPATION

Project T5

Key Metrological Parameters for Lithium-ion Cell Standardization

Quick Start Guide

You are welcome to register here for the testing campaign in which battery parameters will be tested by laboratories worldwide. Interest has already been received from Europe, North America, Asia.

Project Status and Schedule

Sample Shipment	From May 2025
Re-shipment	Until 10th Aug 2025
Data exchange	Until 20th Aug. 2025
Workshop	23/24th Sep. 2025

Participation Fee

There is no participation fee and also no funding. Cells will be distributed by BAM free of charge. Participants fund their own involvement in the project.

Participants´ contribution

Participants choose their contribution from

I) Visual inspection only
II) Electrical cycling tests
III) Impedance/DC-pulse tests
IV) Battery abuse testing

For more information:

Contact: zeteze@bam.de

Project lead: Dr. Anita Schmidt

Dr. Nils Böttcher, Dr. Tim Tichter

Federal Institute for Materials Research and Testing (BAM), Berlin, Germany

Background

R & D aims for optimization of battery and cell characteristics (power, weight, lifetime, costs, safety and sustainability, …). This leads to a large variety of cells and battery types on the market. Even when having the same format, their usage in a device may not always be possible for either functional or safety reasons. Clearly it cannot be the aim to allow only one single cell type, format or chemical composition. Nevertheless it may be advantageous to standardize a limited number of cell types, in particular for small devices such as flashlights, laptops, mobile phones, etc. To date, however, cell characteristics lack in standardization. While industry does already specify formats (e.g. the outer dimensions), it is the task of metrology to provide standardized measurement routines, performance indicators and tolerances and references.

Objective

The aim of this study is to identify, if it is possible to **define parameters** and **minimum requirements** for harmonized /standardized cell types and their testing. To this aim

- An international **round robin campaign** is initiated. Particularly, implications from shipment impacts as well as storage and use of batteries resulting in differences among laboratories will be addressed
- The aim is to develop **reference data and test protocols** for ensuring data quality, identification of tolerances, uncertainty and reproducibility data.

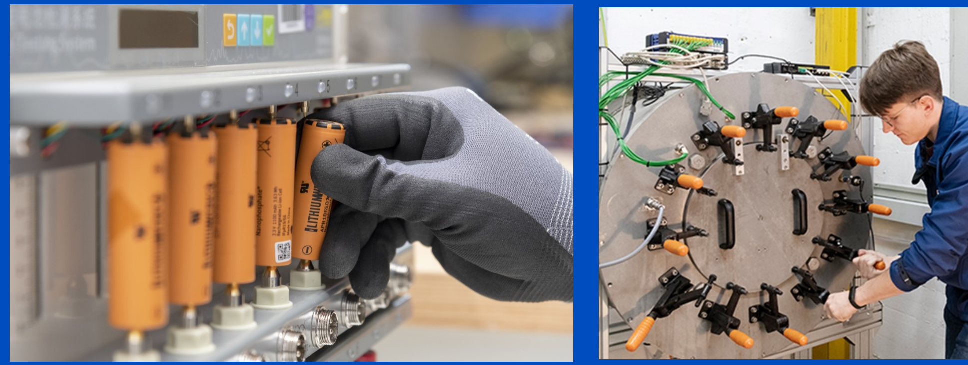

Fig. 1 Lithium-ion cells charging and discharging tests (left) and abuse tests (right)

Testing campaign

The testing campaign consists of four subjects. These are elaborated in detail in the VAMAS-battery testing protocol. Participants are free to choose their contribution.

I) Visual inspection

Comprises an assessment for obvious deviations from appearance of a normal begin-of-life (BOL) cell (cf. figure 2).

Fig. 2 Illustration of the visual inspection of a 18650 cell .

II) Electrical cycling tests

Consists of ten charge/discharge cycles and one steady-state voltage (quasi open circuit potential) measurement with a pre-defined testing protocol (cf. figure 3).

III) Impedance + DC-pulse tests

Electrochemical impedance spectroscopy at a defined state of charge of the cells and DC -pulse tests as performance indicators.

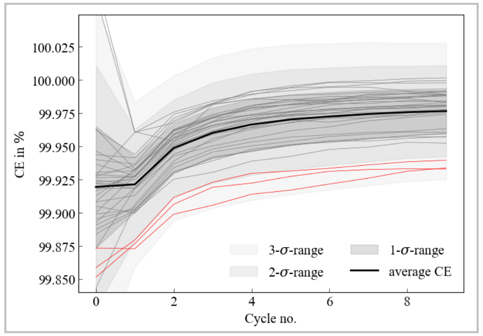

Fig. 3 Tolerances in Coulombic efficiencies of LFP-18650 cells for the VAMAS campaign.

IV) Battery abuse testing

Thermal runaway is deliberately initiated via heating. Parameters for heating rates, positioning of thermocouples, and other details are provided in the protocol.

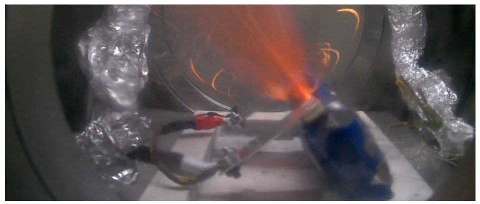

Fig. 4 A snapshot of the moment of thermal runaway failure of a Lithium-Ion cell.

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May 2025

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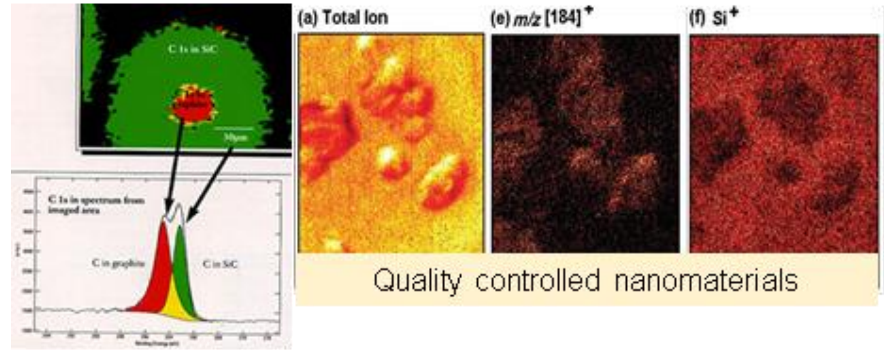
Surface Chemical Analysis

Technical Work Area 2

Call for Interest



Project A46
Spectroscopic Imaging of Nanomaterials by Surface Analysis Methods

Objectives

◦ Determine international interest for standardization of spectroscopic chemical imaging for core/shell nanomaterial characterization.

◦ Developed activities in the project will include identification and preparation of suitable test samples and procedures, determination of measurement repeatability and uncertainties across laboratories.

Background

Mass production of nanomaterials in industrial fields requires a large variety of physicochemical characterizations. One great concern is measuring the chemical bonding within core/shell nanomaterials for quality control and potential degradation with time. Unfortunately, no validated chemical analysis tool and method are available yet for the individual particle detection level. As an alternative, spectroscopic chemical imaging using surface analysis techniques with micrometer resolution is proposed as a potential method for standardized evaluation.
It is anticipated that such methods can provide statistical assessment of the chemical bonding information for core/shell nanomaterials and their quality control.

Standardization Needs

The motivating need of this effort is a methodology for measuring chemical bonding before and after exchange of core/shell nanomaterials, which is a frequent procedure for various industrial purposes. There are several standards for nanomaterial measurement but no validated existing standard method(s) for determining chemical information of core/shell bonding.

The anticipated usage of a documentary standard for these measurements is high, given the large volume of nanomaterial production and related development work worldwide both in academic and industrial organizations

Work Programme

Procedures for assessment of chemical bonding will be developed for several techniques, including:

- Elemental distribution by XPS imaging
- Distribution of molecular fragmentation by TOF-SIMS
- MALDI-TOF
- Soliciting other technique suggestions

Statistical analysis of the results from participants will be conducted as consistent with ISO/TS 5725.

Deliverables and Dissemination

◦ Development of procedures for assessment of chemical bonding on core/shell nanoparticles.
◦ Potential ILC to evaluate developed protocols.

Funding

Participants fund their own involvement in the project.

Status

◦ Independent tests of XPS and TOF-SIMS, and their image analysis were developed and published.
◦ Feasibility tests of the above imaging analysis method are under progress.
◦ Looking for interested partners for an interlaboratory study.

References

Kim, J.W. *et al.*, *Surf. Interface Anal.* **2014**, 46: 193-196. DOI 10.1002/sia.5404
Son, J.G. *et al.*, *Nanoscale* **2016,** 8(8):4573-8. DOI: 10.1039/c5nr07592k

Contact information:

Project Leaders:
Dr. Jeong Won Kim
Korea Research Institute of Standards and Science (KRISS), Korea
jeongwonk@kriss.re.kr

Dr. Tae Geol Lee
KRISS), Korea
tglee@kriss.re.kr

TWA Chair and Vice-Chair:
Prof. Ian Gilmore
National Physical Laboratory, UK
ian.gilmore@npl.co.uk
Dr. Charles Clifford
National Physical Laboratory, UK
charles.clifford@npl.co.uk

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July 2025

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Surface Chemical Analysis

Technical Work Area 2

Call for Participation

Project A47

Evaluation of area selection fidelity in small-area XPS measurements

Background

◦ Evaluate a methodology for determining the selected area with small-area X-ray photoelectron spectroscopy (XPS) utilizing a specified artifact and measurement protocol

◦ Provide input into ISO/TC 201/SC 2 for the further development of ISO 18516 "Surface Chemical Analysis – Determination of lateral resolution and sharpness in beam-based methods with a range from nanometers to micrometers.

Background

Small-area XPS is increasingly used for the evaluation of structures on surfaces but is missing a validated methodology for quality control. Operators may thus be unable to determine if they use an incorrect field-of-view (FoV), such as sampling too large of an area, or a measurement position with an offset from the center of the structure. In such instances, electrons from the structure as well as electrons from the substrate would reach the detector.

To address this problem a test material was developed presenting two types of structure sets: Cr squares and Cr circles with lateral dimensions ranging from 300 µm to 5 µm embedded into an Au-coated substrate. A metrological SEM was used to confirm artifact consistency.

This test material is suitable for most beam sizes used in XPS for analysis of surface structures as small as 5 µm.

Standardization Needs

Both a methodology and calibration artifacts are desired for improving and validating lateral resolution and area selection of XPS to an accuracy of a few micrometers.

This study will provide results for the revision of ISO 18516 in ISO 201/SC2.

Work Programme

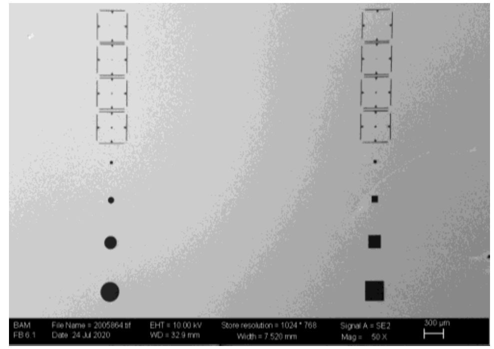
◦ Participants will receive a protocol, test artifact, and a data reporting sheet

◦ Survey and high-resolution measurements with the instrument and software of the participants on each structure with at least three different beam sizes or apertures are demanded.

◦ Statistical analysis of the results from participants will be conducted as consistent with ISO/TS 5725.

Deliverables and Dissemination

Results will be centrally evaluated, with a plan for publication in a peer-reviewed journal. Results will also be used to inform the revision of ISO 18516 ("Surface Chemical Analysis – Determination of lateral resolution and sharpness in beam-based methods with a range from nanometres to micrometres).



An overview SEM image of a test artifact

Participation

Participants need to be skilled operators with access to their own XPS and analysis software.

Funding

Participants fund their own involvement in the project.

Status

This project is identifying participants and readying the distribution of artifacts and the developed protocol.

The protocol and the test material were developed by BAM. The test material has been tested with two different XPS instruments and it was subjected to a quality control with metrological SEM. The uncertainties in length range from 0.05% to 11%.

References

▪ Baer, D. R. et al., *Surface and Interface Analysis* 29.11 (2000): 766-772. ▪ U. Scheithauer, *Surface and Interface Analysis* 40, no. 3–4 (2008): 706–709.
▪ Stockmann, Jörg M., et al, *Surface and Interface Analysis* 57 (2025): 131-136.

For more information:

Jörg M. Stockmann
Federal Institute for Materials Research and Testing (BAM)
joerg-manfred.stockmann@bam.de
Dr. Jörg Radnik
Federal Institute for Materials Research and Testing (BAM)
joerg.radnik@bam.de

TWA Chair and Vice-Chair:
Prof. Ian Gilmore
National Physical Laboratory, UK
ian.gilmore@npl.co.uk
Dr. Charles Clifford
National Physical Laboratory, UK
charles.clifford@npl.co.uk

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July 2025

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Surface Chemical Analysis

Technical Work Area 2

Call for Participation

Project A48

Round robin test of three-dimensional nanoscale roughness artifacts with specified statistic quantities

Objectives

This project aims to verify the quality of the roughness artifacts with specified statistic quantities designed and fabricated by focused ion beam (FIB).

Background

Surface structures are closely related to functional performance of various devices. Quantitative measurements of surface areal roughness are critical in many fields of nanotechnology. Atomic force microscopy (AFM) has long been used in surface topography measurements. However, there is currently no standardized roughness reference materials suitable for AFM. For this purpose, roughness surfaces with specified statistic quantities were designed and fabricated by FIB. The aim of this project is to test these reference materials.

Standardization Needs

Roughness is one of the important values in the field of precision mechanical engineering. However, the AFM roughness measurement procedure cannot be calibrated due to the lack of roughness standards. Through this ILC, the standardization for designing and fabricating roughness surfaces with specified statistic quantities will be established.

Work Programme

Participants will be asked to measure a sample with 12 micro/nano roughness surface patterns with specified statistic quantities. An electronic copy of the original raw data, together with a report with detailed setting parameters and other illustrations should be submitted. The original data will be used to calculate the statistic quantities of the roughness surfaces with the same procedure.

Statistical analysis of the results from participants will be conducted as consistent with ISO/TS 5725.

Deliverables and Dissemination

Results will be used to compare the S_q, S_{sk}, and S_{al20}, and to draft an ISO standard for the development of nanoscale roughness artifacts, submitted to ISO TC 201/SC9 (scanning probe microscopy) for consideration

Funding

Participants fund their own involvement in the project.

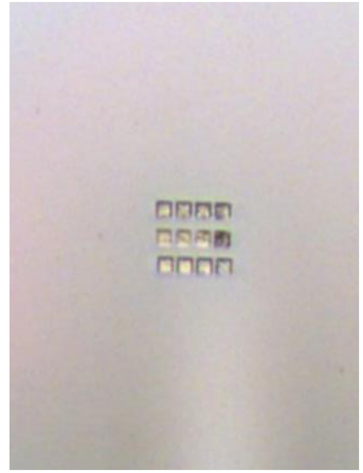

Figure 1. The optical image around the test area of the sample

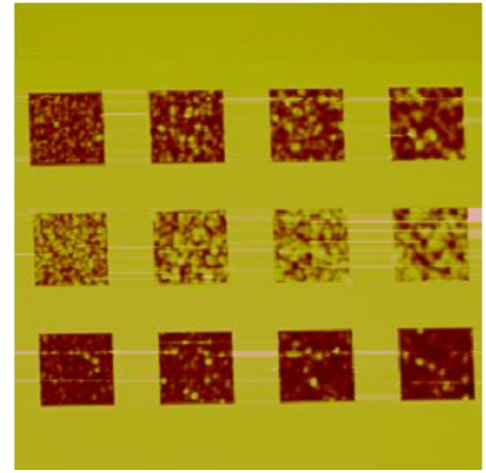

Figure 2. Full-area scan (31 µm × 31 µm) covering all the 12 surfaces.

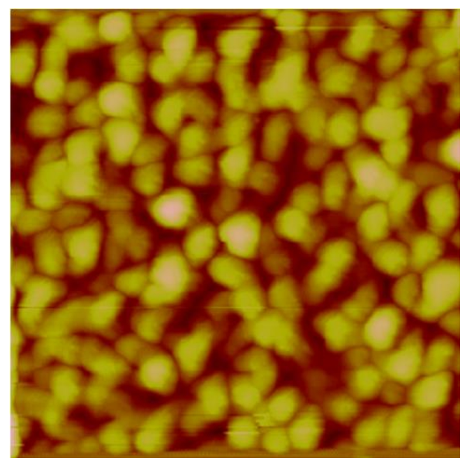

Figure. 3. A scan (5 µm × 5 µm) locating the roughness surface in the image center.

For more information:

Project Leaders:
Dr. Yushu Shi
National Institute of metrology, China.
shiys@nim.ac.cn

Dr. Yuhang Chen
University of Science and Technology of China.
chenyh@ustc.edu.cn

TWA 2 Chair
Prof. Ian Gilmore
National Physical Laboratory, UK.
ian.gilmore@npl.co.uk

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July 2025

Quantitative Microstructural Analysis
Technical Work Area 37

Call for Participation

Project 7
Evaluation method of surface layer quality of TEM specimen prepared by focused ion beam processing

Objectives:

The aim of this Interlaboratory Comparison (ILC) is to develop a method for evaluating damage layers introduced by FIB fabrication on the surface of transmission electron microscopy (TEM) specimens. Such damage layers are commonly affected by the acceleration voltage conditions at the finishing processing stage. A single-crystal Si is provided, and the thickness of the amorphous layer introduced as a results of one of the ionic damages is determined by TEM observation.

Background:

In the case of semiconductor Si devices, an amorphous layer typically forms on the surface during FIB fabrication; in order to evaluate device properties, it is necessary to carry out a finishing process to reduce this amorphous layer. In metals, these finishing processes can introduce fine lattice defects on the surface, which are deleterious for TEM observations. Attempted methods for removing the damage layer have included lessening the acceleration voltage during FIB fabrication, and removal of the damage layer by Ar^+ ion milling. However, a well-defined method for evaluating the area of the damaged layer in FIB fabricated samples has not yet been established.

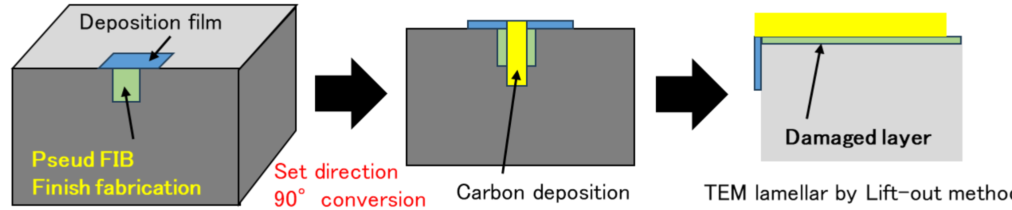
Standardization needs:

In order for TEM specimens prepared by FIB fabrication techniques to accurately reflect the desired material microstructure, the damaged layer must be removed. It is necessary to establish a procedure to objectively evaluate the thickness of the damage layer introduced in various TEM specimens.
Since the thickness of the amorphous layer introduced as a damage layer in Si specimen varies depending on the acceleration voltage and other conditions during FIB processing, a common criterion for evaluating this thickness should be developed. The sample distributed is (001) single crystal Si, and the FIB fabrication conditions are reported in this ILC.. Verify that differences in damage layers caused by differences in FIB equipment and finishing conditions can be evaluated by TEM observation as differences in amorphous layer thickness.

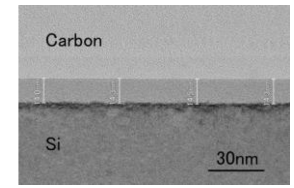
Relevant Standards:

ISO/FDIS 17297 FIB Vocabulary
ISO/TC202/SC1 - in progress

ISO/CD 16887 Guidelines for TEM specimen preparation using FIB
ISL/TC202/SC3 - in progress



I. FIB finishing fabrication has been demonstrated at edge area of [001] single Si crystal.
II. The sample is taken out of the FIB equipment and tilted at 90° to protect the processing surface by C deposition.
III. TEM lamellar is picked up and the thickness of amorphous region at the top of Si wafer is measured.



Work Programme:

1. **June 2025:** Specimens will be delivered to participants along with the protocols.
2. **June/Aug 2025:** FIB processing of thin lamellae and successive TEM observation will be done by the participants in accordance with the protocols provided.
3. **September 2025:** Data submission and analysis
4. **October 2025:** First draft of ISO/PSI will be established.
5. **January 2026:** A peer-review publication is prepared on next step.

Deliverables and Dissemination

- Results disseminated in a peer-reviewed scientific journal.
- Evaluation method of damaged layer of TEM specimen made by FIB will be established by the ISO in the TC 202/SC3.

Funding:

Participants fund their own involvement in the project.

Status: The project is due to start in March 2025 for a duration of up to 24 months, additional participants are welcome.

For more information:

Project Leader:
Dr Masaaki Sugiyama
Osaka University, Japan
sugiyama@mat.eng.osaka-u.ac.jp

TWA Chair
Dr Vasile-Dan Hodoroaba Federal Institute for Materials Research and Testing (BAM), Germany
dan.hodoroaba@bam.de

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May 2025

Raman Spectroscopy and Microscopy

VAMAS

Technical Work Area 42

Project 7

Protocols for relative intensity calibration of Raman spectrometers

Objectives

◦ Obtaining relative intensity calibration for a Raman spectrometer, including polarization dependent response and an estimate of error.

◦ Facilitating the comparison of Raman spectra acquired on different instruments

Background

Dr. Ankit Raj (Gakushuin University, Japan) with the support of National Metrology Institutes has initiated the project "*Method development and validation for relative intensity calibration of Raman spectrometers*" under the VAMAS Technical Work Area (TWA) 42. This project continues the ongoing efforts towards establishing a reliable protocol for relative intensity calibration, and which includes polarization correction as well. This project aims to validate the proposed protocol among the participating laboratories and refine the technique based on the obtained results.

Measurement Methods

Specific protocols involve measuring standard and commonly available liquid samples, together with a broadband light source. Measurement conditions are to be optimized for high SNR data which will be analyzed to extract out the instrument response function.

Standardisation Needs

Raman spectroscopy is undergoing rapid progress in applications relevant to industry, medicine as well as chemical and physics research. However, there is poor comparability between results from different instruments and between different laboratories with regard to the Raman intensities.

The measured Raman intensities are dependent on the incident laser and optical configuration as well as polarization response, which differ from machine to machine. Thus, there is an urgent need to address this variability via establishment of protocols for intensity calibration.

Work Programme

Lead will prepare the broad band light source which will be shipped to the project participants for measurement. Liquid samples (of required quality) will be sourced by the participants. Sets of measurements on these will be made as per the protocol and shared. Data analysis will be made to obtain the instrument response (for parallel and perpendicular polarizations).

Statistical analysis of the results from participants will be conducted as consistent with ISO/TS 5725.

Call for Participation

Sample	Data analysis procedure and outcome
Broadband white light source	Least-squares fit to get first set of sensitivity information (∥ and ⊥ polarizations)
Organic liquids (benzene, cyclohexane, CCl_4, benzonitrile, toluene)	Least-squares fit of band area ratios to get second round of sensitivity information, and Raman depolarization ratios (∥ and ⊥ polarizations) Validation by comparison of relative intensities and/or Raman thermometry

Fig. 1: Proposed protocol for relative intensity calibration under this project

Deliverables and Dissemination

Results of the inter-laboratory study will be disseminated at scientific conferences and peer-reviewed journal article acknowledging the participants with co-authorship of those that contribute with data analysis and manuscript writing.

The results will be used to improve the protocols under study and update two related CWAs, with the final aim of proposing their further standardization.

International Participation

Current participants include organisations from countries from EU, US, UK, Canada, Brazil and Japan. Additional participants are welcome to join.

Funding

Participants fund their own involvement in the project. The lightsource sample holder will be shipped to the participant. Detailed information for the required liquid samples (to be acquired by the participants) will be provided.

Status

The project is due to start on 30th July 2025 and finish by June 2026.

For more information:

Project Leader:
Dr. Ankit Raj
Gakushuin University, Japan
ankit.raj@gakushuin.ac.jp

TWA 42 Chair
Dr. Erlon Henrique Martins Ferreira
Inmetro, Brazil
ehferreira@inmetro.gov.br

Registration link

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July 2025