

News from 21GRD09 MetroPOEM

The third progress meeting (M27) was held at **Danmarks Tekniske Universitet Campus** in Risø, Denmark from 13th-15th November 2024 and was attended by 20 participants in person and a further 25-30 on-line participants, including the stakeholder community.



Participants at the M27 meeting

Project management board meeting

Twenty-two out of the total of 24 members of the Project Management Board participated (13 in person, 9 online) in the meeting. The main points of the meeting were (i) that Lucille Chambon will replace Valérie Lourenço as the CEA PMB member (Valérie is moving to a new position in CEA), (ii) arrangements for the shipping and registration of WP3 samples were discussed in detail, and (iii) Universität Wien has joined the project as a partner, and their technical input was described by Karin Hain.

Project progress meeting

Forty-one persons from the project consortium (22 in person, 19 online) took part. The project work packages were presented by their respective WP leaders; in general, all the technical work packages are on schedule. The main points were:

- WP1: Since the M18 meeting, the materials for measurement have been delivered. Measurements are expected to be complete in March. The interlaboratory comparison report and good practice guide will be written and published by the end of the project.
- WP2: Presentations of ongoing work were presented by JSI, LGC and Hereon. Highlights since the M18 meeting are (i) that recommendations for setting up a high precision measurement protocol are in preparation, and (ii) a new variation of the isotope mixture approach (improving on the gravimetric isotope mixture method) has been developed by PTB and BAM that introduces ion chromatography to determine the elemental mass fractions leading to an approach being independent of the purity of the spike materials, and therefore does not contribute to the uncertainty of the absolute isotope ratios; recommendations for setting up high precision protocols will be provided.

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- WP3: The preparation of the solid and liquid reference materials was described, including work on the dissolution of the prepared solid material. Both the solid and liquid reference materials will be shipped in late November/early December as were some approaches for the dissolution of the solid material. A revised plan was presented for reporting of results and delivery of the documents describing (i) the preparation of the materials, and (ii) the outcome of the inter-laboratory comparison.
- WP4: The water samples from the North Sea were delivered to participants in September; the homogeneity and stability measurements are ongoing, with completion due in November and the evaluation of the results in January 2025. Characterisation of the reference material will commence after sample shipment in November with the completion of analyses in February 2025. The draft report will be completed by the end of May 2025.

Stakeholder meeting

The meeting was chaired by Jixin Qiao, Professor at DTU Sustain. The stakeholder participants were from a range of organisations. Progress in all the project work packages was reported, and the interactions with similar activities were briefly discussed.

Next meeting and upcoming events

The third MetroPOEM stakeholder workshop will be held on 15 May 2025, followed by a meeting of the BIPM CCRI(II) Task Group on Mass Spectrometry (CCRI(II)-MS-TG). Both events are open to all who wish to attend, and will be held in-person at NPL, UK.

Please contact Ben Russell for registration forms, meeting details and information on accommodation at ben.russell@npl.co.uk

The MetroPOEM project will also be represented at these events:

- | | |
|----------------------------------------------------------|---------------------------------------------------------------------------------------|
| European Winter Conference on Plasma Spectrochemistry: | https://www.ewcps.eu/ |
| • Berlin, 2-7 March 2025 | |
| Norwegian Environmental Chemistry Symposium: | https://www.envirochem.no/ |
| • Geilo, 9-12 March 2025 | |
| Congrès International de Métrologie: | https://www.cim-metrology.org/ |
| • Lyon, 11-14 March 2025 | |
| 24th International Conference on Radionuclide Metrology: | https://icrm2025.org/ |
| • Paris, 19-23 May 2025 | |
| One Ocean Science Congress | https://one-ocean-science-2025.org/ |
| • Nice, 3-6 June 2025 | |

The next project management board and project progress meetings will be held on 24 and 25 June 2025 at Türkiye Bilimsel ve Teknolojik Araştırma Kurumu (TÜBİTAK), Kocaeli, Türkiye.

Please contact Oktay Cankur for registration forms, meeting details and information on accommodation at oktay.cankur@tubitak.gov.tr

Special Issue of *Frontiers in Chemistry*: Advances in Stable and Radioactive Isotope Ratios Analysis by Mass Spectrometry

This Research Topic, co-edited by Tea Zuliani (IJS), Johanna Irrgeher (MUL), Dmitriy Malinovskiy (LGC), Norbert Kavasi (QST), Ben Russell (NPL), aims to explore the latest advancements and applications of MS in isotope ratio analysis as well as radionuclide concentrations, highlighting innovative methodologies, case studies, and cross-disciplinary impacts. Emphasising both stable and radioactive isotopes, the contributions will address current challenges and future directions in leveraging MS technology to solve critical problems in environmental monitoring, food safety, and health sciences. We welcome Original Research, Review, and Perspective articles on themes including, but not limited to:

- Novel instrumental development;
- Development of new and improved analytical methods for stable traditional and non-traditional isotope ratios;

- Development of new and improved analytical methods for the determination of concentration and isotope ratios of long-lived radionuclides;
- Application of stable traditional and non-traditional isotope ratios in studies related to environment, food, and health;
- Application of long-lived radionuclides and their isotope ratios in studies related to environment, food, and health.

We look forward to your contributions and collaborations. Let's advance the field of isotope research together. Feel free to reach out to Tea Zuliani (tea.zuliani@ijs.si) if you have any questions.



<https://www.frontiersin.org/research-topics/67498/advances-in-stable-and-radioactive-isotope-ratios-analysis-by-mass-spectrometry>

- Manuscript Summary Submission Deadline 25 February 2025
- Manuscript Submission Deadline 25 June 2025

Science Feature – Advancing stable isotope ratio measurements of environmental pollutants

Contributors: AU/UGOT, BAM, DTU, Hereon, IFE, **JSI**, LGC, LNE, LUH, MUL, NPL, PTB, TUBITAK

The study of metal isotopic abundance variations in biological and geological systems is a dynamic and multidisciplinary field with far-reaching applications in areas such as inorganic chemistry, environmental science, climate research, biomedicine, archaeology, biology, and mineral exploration geochemistry. This growing interest is fuelled by advancements in state-of-the-art analytical techniques, which enable the precise determination of isotopic compositions across a wide range of natural and anthropogenic materials, opening new frontiers in scientific discovery and innovation. Precise and accurate isotope ratio measurements are the essential cornerstone for advancing research and applications in this field.

In Work Package 2 (WP2) of the MetroPOEM project, we are developing new and improved methods for stable and long-lived radioactive isotope ratio measurements. Based on mass spectrometry, these methods aim to achieve uncertainties that resolve natural mass-dependent isotope fractionation. The project focuses on selecting environmentally relevant elements (Li, B, Cr, Cd, Ni, Sb, Pb, and U) as key indicators for developing and optimizing isotope ratio measurement procedures.

To obtain accurate and precise isotopic ratios, the samples need to be properly prepared. Several steps are usually applied before isotope ratio measurement. Depending on the type of the sample, there may be pre-treatment steps needed. For solid samples, it consists of a digestion or extraction, while for liquids, usually a preconcentration is needed. Next is the separation step to isolate the element of interest from the sample matrix, and finally, the determination of the isotope ratios by measurement of the isotope responses and correction for instrumental isotopic fractionation (IIF, Figure 1).

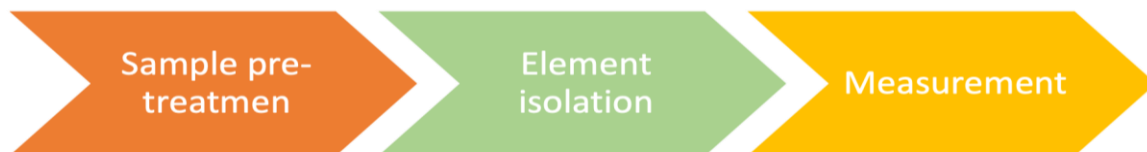


Figure 1: Schematic of the isotope ratio determination procedure.

A range of instrumental techniques are employed, including multi-collector ICP-MS (MC-ICP-MS), sector field ICP-MS, quadrupole ICP-MS, and ICP-MS/MS. The performance of these techniques was assessed and compared in terms of accuracy and precision (Figure 2).

For this purpose, the isotopic reference materials (IRM) were used. These materials are limited to very few elements. They are needed to calibrate mass spectrometers, that is, to correct for the instrumental isotopic fractionation (IIF) during mass spectrometric measurements. During the compilation of the list of available IRMs we found out that for certain elements' IRMs are not anymore available, as is the case for Ni (NIST SRM 986) or they are completely missing (Sb).

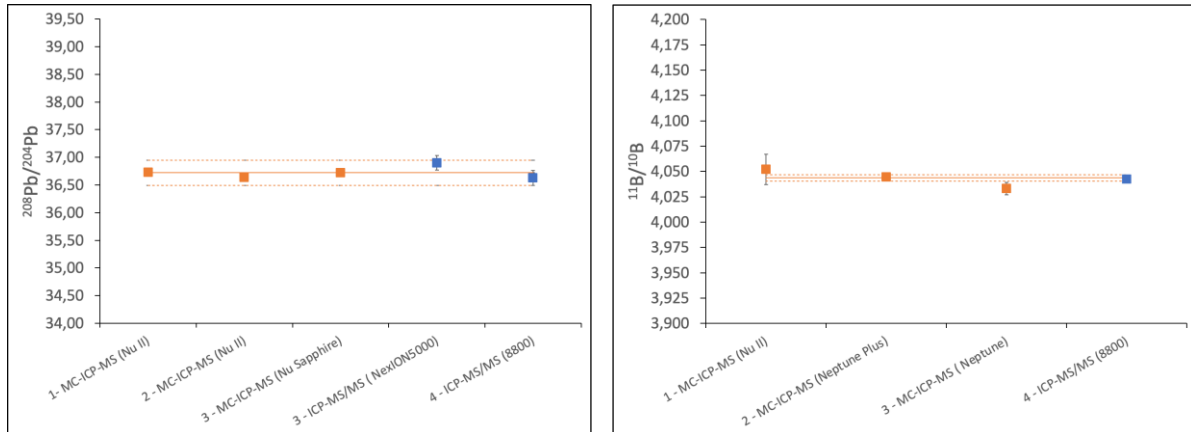


Figure 2: Comparison of isotope ratios for selected elements obtained with different mass spectrometers.

A significant challenge in mass spectrometry, particularly in inductively coupled plasma mass spectrometry (ICP-MS) is instrumental isotope fractionation (IIF). When unaccounted for, this phenomenon leads to biased isotope ratio measurements. Traditionally, IIF has been corrected using empirical equations such as Power, Exponential and Russel law. However, a few studies show inaccuracy or significant deviations from true values when applying these conventional models. In the frame of WP2, we are improving current and developing new approaches based on inter-element normalisation and without *a priori* assumptions. PTB and BAM optimised the isotope mixture approach, a primary method for the determination of SI-traceable isotope ratios. The latest variation of the approach introduces ion chromatography to render the knowledge of the elemental mass fractions in the enriched isotope solutions superfluous. This way it significantly simplifies the classical isotope mixture approach. This new approach has been published by Flierl *et al.* (2024, <https://doi.org/10.1007/s00216-024-05465-9>) and it will be applied in the interlaboratory comparison on Li isotope ratios (CCQM-K182/P233) for testing the achievable uncertainty level and the comparability of laboratory results.

Although the primary method is a strong asset, for routine use, the method has, unfortunately, some throwbacks like enriched isotopes are not available for many stable elements, and/or the required uncertainty level does not justify the costs and efforts of applying this method. Therefore, additional methods are required. For the isotopic systems where the gravimetric isotope mixture cannot be applied, and certified iRM are not available, modelling of the IIF is being tested. Another method that is being optimised is the regression method to correct for instrumental isotope fractionation. The method is based on simultaneous measurement of analyte, for which isotope ratios are to be determined, and internal standard with known isotope ratios. In this case, the IIF correction factor can be accurately determined for the internal standard from its measured and *a priori* known isotope ratios and then be extrapolated to the analyte.

Lab Training for ICP-MS: Workshop on radioanalytical chemistry for environmental monitoring, nuclear decommissioning, emergency preparedness, radioecology and tracer studies

Jixin Qiao, DTU

The **NKS-B RadWorkshop 2024**, held from 9-13 September 2024, at the DTU Risø Campus in Roskilde, Denmark, brought together 104 participants from nearly 60 organizations in 20 countries to

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focus on advancements in radioanalytical chemistry and its application in various fields. The audiences were a good mixture of all parties involved in the field including regulators, operators, service partners, researchers, young scientists and students. This international gathering provided a unique platform for knowledge exchange and professional development. The workshop's program was designed to address key challenges and innovations in radiochemistry, and was divided into 2 parts:

Lectures (3 days) given by 10 invited professionals and 24 seminar participants. 7 posters were also presented at the workshop. The presentations covered a broad spectrum including theoretical principles of radiochemistry and measurement techniques (α , β and γ), recent advancement in separation techniques, materials and detection instruments. Specific highlights included ultra-trace analyses with AMS, optimizing ICP-MS/MS for radionuclide measurement, applications of radioanalytical chemistry in nuclear decommissioning, environmental tracer applications and emergency preparedness.

Laboratory training (2 days) by experienced professionals, with nearly 45 participants engaging in this hands-on session. The lab training focused on the radiochemical analysis of ^{90}Sr and advanced measurement techniques using ICP-MS/MS, as well as liquid scintillation counting (LSC) & beta counting. The ICP-MS/MS lab training session was supported by **MetroPOEM project**. This session provided participants with practical skills and in-depth knowledge on advanced analytical techniques, specifically in the use of triple quadrupole ICP-MS.



Workshop participants

Short reports from the technical work packages.

WP1: NPL

The starting materials for the inter-laboratory comparison in WP1 have been fully characterised and homogeneity and stability testing will continue throughout the project. Progress of WP1 has been impacted by delays in dispatching the materials to the participating laboratories through getting the appropriate agreements and activity levels for all participating labs in place. This has now been resolved and labs have received a range of single and mixed radionuclide standards for concentration and isotopic ratio measurements depending on what their labs are able to receive. Measurement of these standards is now underway and expected to be completed in March 2025, which will be used as the basis of the interlaboratory comparison report.

WP2: IJS

The work on the development and optimisation of new and/or enhanced separation methods for selected elements is being finalised.

Currently, the focus is on the development of instrumental isotope fractionation corrections. The isotope mixture approach, a primary method for SI-traceable isotope ratios, has been improved with the introduction of ion chromatography to determine the elemental mass fractions in the enriched isotope solutions. It significantly simplifies the classical isotope mixture approach. However, its limitations necessitate alternative approaches, such as IIF modelling and regression-based corrections. New methods aim to enhance accuracy, affordability, and practicality, supporting broader applicability in isotope ratio measurement systems.

WP3: CEA

The radionuclide stock solutions were collected during the summer and CEA and ČMI were able to proceed with the production of the two reference material candidates in September and October 2024 respectively. For the liquid materials, about 40 L of sterilised and labelled

seawater was produced and the 0.5 L samples will be sent to the intercomparison participants in November 2024. The solid material was synthesised and dried in September (Figure 3), then ground in a jar miller, sieved to 250 µm (Figure 4) and bottled in October. Preparations are now underway to ship the 100 g samples, which will be sent to the intercomparison participants from 19 November 2024. In the meantime, to prepare for the solid material intercomparison, participants tested their dissolution procedures on the inactive solid samples shipped in spring 2024.



Figure 3: A tray of the solid material as it gels (left) and then dries (right).



Figure 4: Handling of the material in a disposable glovebox after jar milling

WP4: TŪBITAK

The certification of seawater CRM for supporting the validation of analytical methods, proficiency testing and thus ensure quality control for the measurement of stable isotopes

WP5: NMBU

Since the last newsletter, the training course for the project as part of the upcoming RadWorkshop 2024 was held at DTU in September, and the project was promoted at the 10th International Conference on Nuclear and Radiochemistry in August. A workshop is planned (by LGC) at the 20th European Winter conference on plasma spectrochemistry. Abstracts have been submitted to the Congrès International de Métrologie (March 2025), the 24th International Conference on Radionuclide

is in progress. The homogeneity and the stability samples had been sent to the participating laboratories in September 2024 and the measurements continues. The study protocol for characterisation has been sent to the laboratories together with the registration form for their participation in the ILC for the measurement stable isotopes. The characterisation samples to the participating laboratories will be dispatched in the first week of December 2024. The characterisation studies are expected to finish by the end of February 2025.



Figure 5: The candidate Seawater CRM for isotope ratio of stable isotopes



Figure 6: The candidate Seawater CRM batch of 470 bottles which are stored at +4 °C

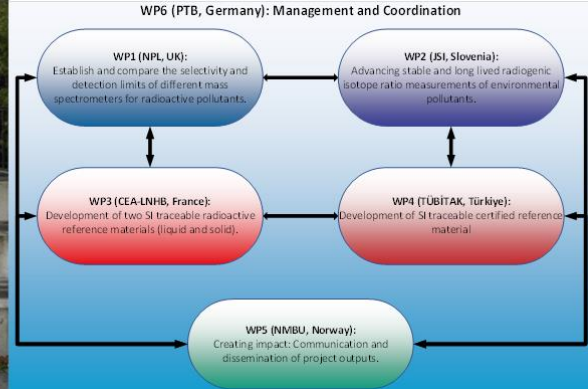
Management and dissemination.

Metrology and its Applications (May 2025) and One Ocean Science Congress (June 2025); the abstracts for CIM 2025 and ICRM 2025 have been accepted.

WP6: PTB

The updated DCE plan has been approved by EURAMET, as have the M18 progress report and the updated data management plan. The project documentation has been updated to include the new project partner, Universität Wien.

MetroPOEM, coordinated by the Physikalisch-Technische Bundesanstalt of Germany, is delivered by a consortium of 24 partners from 14 countries.



Physikalisch-Technische Bundesanstalt (Coordinator, WP6 leader)

Bundesanstalt für Materialforschung und -prüfung
Commissariat à l'énergie atomique et aux énergies alternatives (WP3 leader)
Český Metrologický Institut
Institut Jožef Stefan (WP2 leader)
Laboratoire national de métrologie et d'essais
Säteilyturvakeskus
Türkiye Bilimsel ve Teknolojik Araştırma Kurumu (WP4 leader)
Aarhus Universitet
Danmarks Tekniske Universitet
Helmholtz-Zentrum Hereon GmbH
Helmholtz-Zentrum Dresden - Rossendorf e.V.
Institut for energiteknikk
Institutul National de Cercetare-Dezvoltare pentru Fizica si Inginerie Nucleara 'Horia Hulubei'
Gottfried Wilhelm Leibniz Universität Hannover
Montanuniversität Leoben
Norges miljø- og biovitenskapelige universitet (WP5 leader)
Helsingin Yliopisto
Institut za nuklearne nauke Vinča Institut od nacionalnog značaja za Republiku Srbiju,
Univerzitet u Beogradu
Göteborgs universitet
Universität Wien
Eidgenössische Technische Hochschule Zürich
LGC Limited
NPL Management Limited (WP1 leader)

Collaborators

Triskem International
Fizinių ir technologijos mokslų centras
Spiez Laboratory

PTB	Germany
BAM	Germany
CEA	France
ČMI	Czechia
IJS	Slovenia
LNE	France
STUK	Finland
TÜBİTAK	Türkiye
AU	Denmark
DTU	Denmark
Hereon	Germany
HZDR	Germany
IFE	Norway
IFIN-HH	Romania
LUH	Germany
MUL	Austria
NMBU	Norway
UH	Finland
VINS	Serbia
UGOT	Sweden
UNIVIE	Austria
ETHZ	Switzerland
LGC	United Kingdom
NPL	United Kingdom
Triskem	France
FTMC	Lithuania
LS	Switzerland

Project information

The overall deliverables and dissemination routes shown in the diagram below
Additionally, the project has an internet presence at:
Project website: <https://www.npl.co.uk/euramet/metropoem>
Linkedin: <https://www.linkedin.com/in/metropoem-project-308762251/>
Research gate: <https://www.researchgate.net/profile/Metro-Poem>
MetroPOEM can be contacted through the project website, or at this email address: metropoem@nmbu.no

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