Metrology for the harmonisation of measurements of environmental pollutants in Europe Newsletter 05 – Summer/Autumn 2025

JRP EPM 21GRD09 MetroPOEM Coordinator: Dirk Arnold (PTB) E-mail: dirk.arnold@ptb.de

The final 21GRD09 MetroPOEM Newsletter



This is the final newsletter of the 21GRD09 MetroPOEM project, coming after 3 years of the project, where the project partners contributed 320 person-months of work. The project has been challenging, but has been ultimately successful, delivering outputs that will address the overall aims of the project, which were to bridge the gap between radiometric techniques and mass spectrometry for the characterisation and detection of polluting long lived radionuclides and stable elements and element tracers by comparing and linking both techniques, thus significantly improving measurement uncertainties and detection limits.

Specific objectives.

- To establish and compare (inter-laboratory) the selectivity and detection limits of different types of mass spectrometers (e.g., AMS, HR-ICP-MS, ICP-MS/MS, ICP-QMS, MC-ICP-MS, SIMS, SNMS, TIMS) for selected radioactive pollutants (e.g., U, Np, Pu, Am) using isotope reference materials and/or activity standards. This includes assessing relative instrument performance with respect to current measurement challenges and establishing detection limits in relation to regulatory waste criteria levels or environmental regulations (WP1).
- To develop measurement methods for isotope ratios that are traceable to the SI by using multicollector ICP-MS and apply these methods on more commonly available techniques (ICP-MS/MS, ICP-QMS) by providing suitable operating procedures focussing on stable polluting elements (e.g., Li, B, Cr, Cd, Ni, Sb, Pb, U). To produce recommendations for sample processing, treatment, uncertainty budgets, and if feasible, the quantification of the so-called mass bias (WP2).
- To develop two radioactive reference materials with the sample matrix containing radioactive pollutants (e.g., U. Np. Pu. Am) for use in an inter-laboratory comparison employing techniques used in objective 1, which will demonstrate the variations in parameters including detection limits, sample preparation requirements, sample introduction methods, total procedural time. and uncertainty budgets (WP3).
- To implement and validate the methods for isotope ratio measurements established in objective 2 by the development of one aqueous certified reference material that is certified for the same stable polluting elements with lowest possible uncertainties using multi-collector instruments, in order to facilitate the calibration of single collector ICP-MS, instrument validation, as well as quality control (WP4).
- To facilitate the take up of the technology and measurement infrastructure developed in the project by the measurement supply chain (e.g., accredited laboratories), standards developing organisations and international organisations (JRC, CIPM CCs [CCQM-IRWG, CCQM-IAWG, CCRI], IAEA, ICRM) and end users (e.g., environmental monitoring agencies) (WP5).

Deliverables and tangible project outputs

The project deliverables were:

- D1: Inter-laboratory comparison report describing low-level radioactive pollutants by different types of mass spectrometers.
- Good Practice Guide on the use of mass spectrometry for low-level radioactive pollutants D2: detection.
- Report describing the development of measurement methods for isotope ratios that are D3: traceable to the SI and summarising the advantages/disadvantages of applying them on more

Page: 1/15





















Newsletter 05 – Summer/Autumn 2025

JRP EPM 21GRD09 MetroPOEM Coordinator: Dirk Arnold (PTB) E-mail: dirk.arnold@ptb.de

- commonly available techniques by providing suitable operating procedures focussing on stable polluting elements.
- D4: Good Practice Guide on sample processing, treatment, uncertainty budgets, and if feasible, the quantification of the so-called mass bias.
- Document describing two radioactive reference materials, developed and shipped to D5: participants for their radioactive content characterisation.
- D6: Inter-laboratory comparison report, describing detection limits, sample preparation requirements, sample introduction methods, total procedural time, and uncertainty budgets.
- D7: Report describing the development of one aqueous certified reference material that is certified for the same stable polluting elements with lowest possible uncertainties.

Additionally, a set of training videos has been prepared, and these videos, along with the project deliverables, scientific presentations and links to peer reviewed papers are all available from the project website: https://www.npl.co.uk/euramet/metropoem

The project has featured as a EURAMET good news story:

https://www.euramet.org/publications-media-centre/news/news/partnership-project-delivering-firstreference-materials-for-stable-isotope-ratios-in-seawater

Final Project Meeting

The final project management board meeting and project progress meeting (M33) were hosted by Türkiye Bilimsel ve Teknolojik Araştırma Kurumu in Istanbul and TÜBİTAK UME at Gebze Kocaeli, Türkiye from 24-25 June 2025 and was attended by 24 participants in person and a further 15 on-line participants.



Participants at the M33 meeting

Page: 2/15



















Newsletter 05 - Summer/Autumn 2025

JRP EPM 21GRD09 MetroPOEM Coordinator: Dirk Arnold (PTB) E-mail: dirk.arnold@ptb.de

Third MetroPOEM stakeholder workshop



The third MetroPOEM stakeholder workshop was held on 15 May 2025, followed by a meeting of the BIPM CCRI(II) Task Group on Mass Spectrometry (CCRI(II)-MS-TG), the meetings were jointly titled Current and future applications of mass spectrometry (https://www.npl.co.uk/events/applications-ofmass-spectrometry). Both events were open meetings, and were held in-person at NPL, UK. There were approximately 50 in person and 120 on-line participants. The agenda was:

Welcome

Introduction to MetroPOEM Introduction to WP1

Preparation of radionuclide standard solutions and progress on interlaboratory comparison exercise

Multi-disciplinary Applications of Mass Spectrometry Techniques in Environmental Radioactivity Research

Introduction to WP2

A novel way to determine absolute isotope ratios by combining mass spectrometry and ion chromatography Investigation of Instrumental Isotopic Fractionation in ICP-MS

Refining Sample Preparation: Optimised Automation for Isotopic Analysis via MC-ICP-MS

Good Practice Guide on sample processing, treatment, uncertainty budgets

Online LC separation as a technique to mitigate ICP-MS interferences: example trace ²³⁹Pu in the presence of U **Introduction to WP4**

Seawater sampling and multi element analysis of matrix rich

Planning and processing of the candidate certified reference material

Development of an aqueous certified reference material certified for stable elements

Introduction to WP3

Preparation of radioactive silica RM Preparation of radioactive seawater RM

Introducing CCRI(II)-TG-MS

Richard Barker, Head of the Energy and Environment Challenge, NPL, UK Dirk Arnold, PTB, Germany Ben Russell, NPL, UK Ben Russell, NPL, UK

Jixin Qiao, DTU

Tea Zuliani, JSI, Slovenia Lukas Flierl, PTB, Germany

Pranav Seena Prem, BAM, Germany

Johanna Irrgeher, MUL, Austria and Daniel Pröfrock, Hereon, Germany Dmitriy Malinkovsky, LGC, UK

John Entwisle, LGC, UK

Oktay Cankur, TÜBİTAK, Türkiye Daniel Pröfrock, Hereon, Germany

Betül Ari, LGC, UK/ TÜBİTAK, Türkiye

Oktay Cankur, TÜBİTAK, Türkiye

Lucille Chambon, CEA, France Lucille Chambon, CEA, France Monika Mazánová, ČMI, Czech Republic

Lisa Karam, NIST, USA

The presentations made at the workshop are available from the MetroPOEM website.



















Metrology for the harmonisation of measurements of environmental pollutants in Europe Newsletter 05 - Summer/Autumn 2025

JRP EPM 21GRD09 MetroPOEM Coordinator: Dirk Arnold (PTB) E-mail: dirk.arnold@ptb.de

Summary of project achievements



Work Package 1: Establish and compare the selectivity and detection limits of different mass spectrometers

Mass spectrometry is increasingly being applied to measurement of medium and long-lived radionuclides as a rapid and sensitive alternative to some decay counting techniques. The availability of suitable standards will support this and provide increased confidence in measurement. The main objective of WP1 was to prepare single and mixed radionuclide standards for a comparison exercise, to compare the capabilities of different mass spectrometry instruments.

A series of single and mixed radionuclide standards were prepared, consisting of various actinides (234U, ²³⁶U, ²³⁷Np, ²³⁹Pu, ²⁴⁰Pu, ²⁴¹Am, mixed ^{239/240}Pu and U_{nat}) and ⁹⁰Sr. These were prepared from traceable standards at the National Physical Laboratory. All standards were characterised for activity level and stable and radioactive impurity content and diluted to the appropriate activity concentrations based on regulatory limits and feedback from the participating laboratories.

Thirteen laboratories have submitted their results for the interlaboratory comparison exercise using a range of mass spectrometric and decay counting techniques. The processing of the data is complete, with results generally showing a good agreement between the submitted values and NPL reference values for single radionuclide standards. All radionuclides were measured using multiple techniques, with inductively coupled plasma mass spectrometry the most common.

Due to the delays with sending the samples or the nature of the samples prepared, there were not results for all techniques, with nothing submitted for thermal ionisation mass spectrometry, secondary ionisation mass spectrometry, or sputtered neutral mass spectrometry. In some cases, the activity levels prepared were deemed too challenging to measure using mass spectrometry. This led to one lab being sent a higher activity level for the radionuclides of interest, whilst in some cases measurements were made using decay counting techniques, or a detection limit for mass spectrometry was reported.

The low activity levels were a balance between preparing standards that some participating laboratories were able to receive with their limits, balancing this against national and international regulatory limits, and levels that would provide useful information on the relative advantages and limitations of different mass spectrometer designs.

Results were submitted for all radionuclides using multiple techniques. Of the radionuclides measured, ⁹⁰Sr had the fewest results and was most measured by decay counting, although ICP-MS and AMS were proven to be effective for low-level measurement of this radionuclide. This radionuclide has the

Page: 4/15





















Newsletter 05 – Summer/Autumn 2025

shortest half-life of those included in the comparison and is therefore the most challenging to measure

by mass spectrometry. The highest number of results were received for single Pu isotopes and ²⁴¹Am.

The results have been written up as a draft comparison report, which shows the relative performance of mass spectrometry for low level radionuclide measurement. Despite the low activity levels prepared, multiple laboratories demonstrated that accurate measurement by mass spectrometry is possible. The results from the comparison exercise are being supported by the second WP1 deliverable- a guide on radioactivity measurement by mass spectrometry, for which the measurement of reference materials in WP3 provided valuable input. A further outcome of Work Package One is that traceable radioactive standard solutions suitable for calibration of mass spectrometry instruments are now available, as well as proving there is a current and ongoing need for the development of these standards and comparison exercises.

Three papers have also been prepared that are linked to WP1. The first is the work of the Consultative Committee on Ionising Radiation (CCRI) Section II Mass Spectrometry Task Group, which has been accepted for publication as part of the International Conference on Radionuclide Metrology (ICRM) Technical Series. Two separate papers have been submitted to the Journal of Analytical Atomic Spectrometry following reviewer comments, both on the characterisation of reaction cell gases to improve interference removal that otherwise impacts radioactivity measurement. One paper characterised methyl fluoride, the second a combination of methyl fluoride and ammonia.

Work Package 2: Advancing stable and long-lived radiogenic isotope ratio measurements of environmental pollutants

Accurate chemical measurement is the cornerstone of reliable scientific data. Whether in environmental monitoring, geosciences, archaeology, or biomedical research, the value of a measurement lies not only in the number itself but in the confidence that can be placed in it. Achieving such confidence requires careful calibration against appropriate standards, thorough understanding of the processes that generate and influence the signal, and transparent evaluation of measurement uncertainty.

Within this broader framework, isotope ratio determination has emerged as a particularly powerful analytical tool. From tracing biogeochemical cycles and dating geological samples, to investigating human migration patterns and ensuring food authenticity, isotope ratio analysis provides insights across an impressive range of disciplines. However, the precision demanded by these applications is high, often requiring the detection of minute variations in isotopic composition.

A crucial prerequisite for obtaining such precision is the effective separation of the analyte isotopes from the sample matrix and from concomitant elements. Without this step, matrix-induced interferences and spectral overlaps can distort results and compromise measurement reliability. Separation procedures not only reduce systematic errors but also minimise the impact of instrumental isotopic fractionation (IIF), ensuring that the isotope signals detected truly reflect the composition of the element of interest.

Advances in mass spectrometry, especially the transition from thermal ionisation mass spectrometry (TIMS) to multi-collector inductively coupled plasma mass spectrometry (MC-ICP-MS), have dramatically expanded the scope and accuracy of isotope ratio measurements. At the same time, the rise of single-collector ICP-MS instruments has created opportunities for laboratories to access isotope data at lower cost, albeit with challenges in precision. To address these challenges, robust methods for correcting IIF remain essential, and the search for the most reliable correction strategies continues to be a topic of active discussion within the scientific community.

In WP2 of the MetroPOEM project, we addressed these points, especially the separation methods and IIF corrections. WP2 focused on advancing measurement capabilities for stable and long-lived radiogenic isotope ratios. The central aim was the development and optimisation of generic, highaccuracy mass spectrometry-based methods for key environmentally relevant elements: lithium (Li), boron (B), chromium (Cr), cadmium (Cd), nickel (Ni), antimony (Sb), lead (Pb), and uranium (U). These elements have been selected due to their environmental significance, regulatory relevance, or potential as markers of geochemical pathways and anthropogenic processes.

Several separation methods are used in analytical chemistry to address interferences, each with its own strengths and limitations. These approaches range from simple mathematical corrections to chromatographic separations and advanced chemical resolution technologies. For the isolation of elements from the seawater matrix, as applied in the MetroPOEM project, a variety of resins and techniques were tested depending on the target analyte. For Li, AG50W-X12 resin was used. For B, approaches included sublimation, AG50W-X8, Amberlite IRA 743, and micro sublimation. Chromium

















JRP EPM 21GRD09 MetroPOEM **Coordinator: Dirk Arnold (PTB)**



Newsletter 05 – Summer/Autumn 2025

was isolated using BioRad AG 1-X8, while Ni was treated with Chelex 100 chelating resin, Ni-Spec, and other methods. Cadmium was separated with AG 50W-X8 and BioRad MP-1M, whereas Pb required a broader range of resins, including Sr-Spec, DGA resin, Pb Resin (Triskem), and BioRad AG1-X8 for anion exchange. Uranium was also isolated by anion exchange using BioRad AG1-X8. In addition to manual methods, automated isolation procedures were developed with the seaFAST and prepFAST systems (ESI), offering higher efficiency and reproducibility in sample preparation.

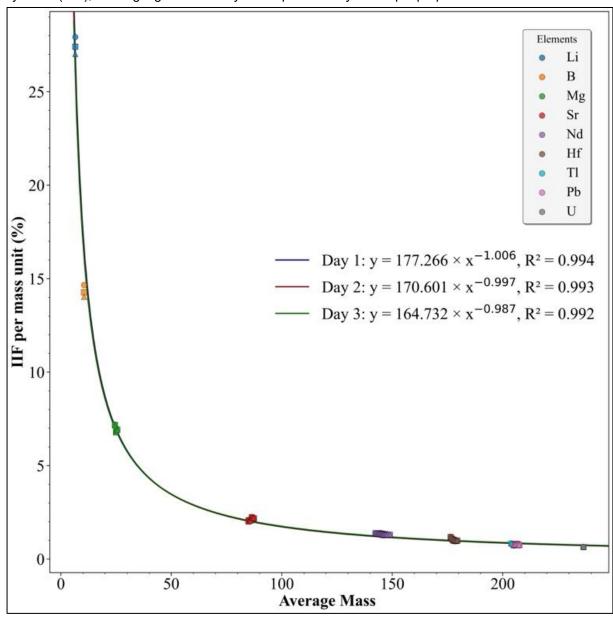


Figure 1: Instrumental isotopic fractionation observed in a Thermo Fischer Scientific Neptune Plus MC-ICP-MS

IIF is a significant challenge in mass spectrometry, particularly in MC-ICP-MS. This phenomenon can lead to biased isotope ratio measurements, necessitating the development of correction methods. 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 aimed to improve current and develop new approaches based on interelement normalisation and without a priori assumptions. During the project the isotope mixture approach, a primary method for the determination of SI traceable isotope ratios, was optimised. The latest variation of the approach introduced ion chromatography to render the knowledge of the elemental mass fractions in the enriched isotope solutions superfluous. This way it significantly

Page: 6/15













Newsletter 05 – Summer/Autumn 2025

JRP EPM 21GRD09 MetroPOEM Coordinator: Dirk Arnold (PTB) E-mail: dirk.arnold@ptb.de

simplifies the classical isotope mixture approach. Another way of correcting for IIF explored during the project was the regression method to correct for instrumental isotope fractionation. The method works by measuring the analyte alongside an internal standard with known isotope ratios. Any difference between the measured and true values of the standard reveals the correction factor, which can then be applied to the analyte for more accurate results. In WP2 we also explored a modelling method (Figure 1) that can be applied when the gravimetric isotope mixture cannot be applied and iRM are not available.

The work carried out in WP2 of the MetroPOEM project demonstrates how the combination of carefully optimised separation techniques and robust IIF correction strategies can significantly enhance the accuracy and SI-traceability of isotope ratio measurements. While chromatographic and automated separation procedures provide powerful tools for isolating analytes from complex matrices, innovative correction models, ranging from isotope mixture techniques to regression-based and modelling approaches, offer promising pathways to overcome the limitations of conventional IIF corrections.

For a more comprehensive overview of the methods developed and optimised within MetroPOEM, as well as the IIF strategies tested, readers are referred to Deliverable D3 - Report describing the development of measurement methods for isotope ratios that are traceable to the SI and summarising the advantages and disadvantages of applying them to more commonly available techniques by providing suitable operation procedures focusing on stable polluting elements and Deliverable D4 -Good Practice Guide on sample processing, treatment, uncertainty budgets, and the quantification of the so-called mass bias. These resources provide in-depth insights and practical guidance to support the wider adoption of best practices in isotope ratio analysis.

Work Package 3: Development of radioactive reference materials

In work package 3, two radioactive reference materials were developed, one solid and one liquid, comprising ²³⁴U, ²³⁵U, ²³⁶U, ²³⁷Np, ²³⁸U, ²³⁹Pu, ²⁴⁰Pu and ²⁴¹Am. Targeted mass fractions for each radionuclide were chosen at the beginning of the project, as well as desired uncertainties (1 % to 5 %)

Both reference materials were obtained by spiking a matrix with well-characterised starting solution of individual radionuclides or a mixture of isotopes of the same element: U, Pu, Am and Np (Figure 2). The starting solutions were characterised with mass spectrometry and radiometric techniques, before being first mixed in HNO₃ 3 mol/L to form a 'multi-RN spiking mixture', which was also characterised.

Seawater was used as a matrix for the liquid RM, while the 'multi-RN spiking mixture' was added to two silica precursors for the solid RM. These silica precursors reacted by hydrolysis and condensation processes (sol-gel), and formed a solid tri-dimensional network with embedded radionuclides. The solid was ground to yield a synthetic sand-like material. The synthesis was adapted from Ref.

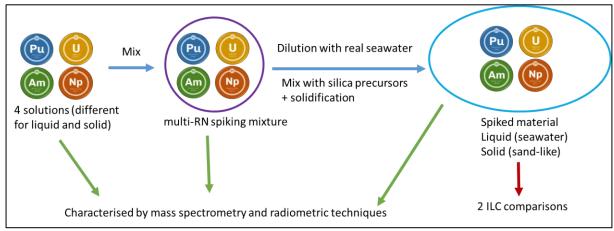


Figure 2. Schematics of the preparation of the radioactive reference materials.

Both reference materials were bottled into around 80 individual containers (500 mL for the liquid and 100 g for the solid) which constituted individual units of the reference materials (Figure 3).

Following the guidelines of ISO 33405, the homogeneity and stability of both reference materials were assessed. In fact, it is important to ensure that the material contained in one bottle is equivalent to that contained in another (between-bottles homogeneity); that sub-samples taken from one bottle are equivalent to one another (within-bottles homogeneity); that there is no variation in the properties of the











Newsletter 05 - Summer/Autumn 2025

JRP EPM 21GRD09 MetroPOEM Coordinator: Dirk Arnold (PTB) E-mail: dirk.arnold@ptb.de

materials during transport (short-term stability); and that there is no variation of the properties of the materials after a long period of time (long-term stability).



Figure 3. Pictures of individual units of both reference materials.

The preparation of both reference materials and the scheme for homogeneity and stability testing are described in deliverable D5, published in May 2025.

Homogeneity measurements were performed with mass spectrometry (238 U and 239 Pu) and 239 Pu and 239 Pu) on both reference materials. Analysis of variance was performed to calculate the variance coming from within the bottles and from between the bottles. The results of the measurement of 238 U and 241 Am for each sub-sample are presented in *Figure* Figure 4, for the liquid (a and b) and the solid (c and d) reference materials.

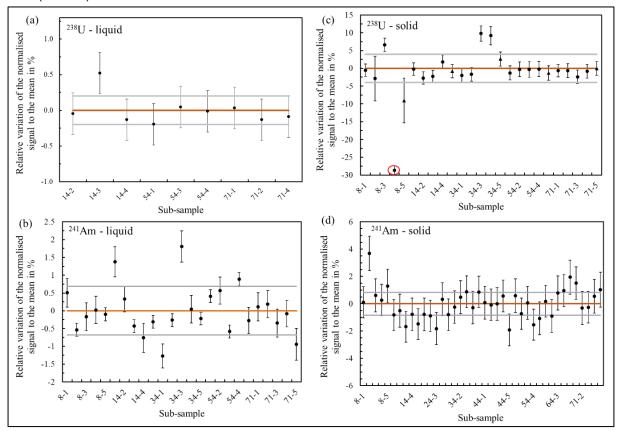


Figure 4. Relative variation of mass spectrometry measurements of sub-samples for 238 U (a and c) and γ -ray spectrometry measurements of sub-samples for 241 Am (c and d)

Page: 8/15



E-mail: dirk.arnold@ptb.de



Metrology for the harmonisation of measurements of environmental pollutants in Europe

Newsletter 05 – Summer/Autumn 2025

In figure 4, the vertical bars represent the uncertainty of each individual measurement, at k = 1, with the orange line representing the average of the values of the sub-samples, and the grey lines represent the standard deviation among the values of the sub-samples. Details on the homogeneity assessment for both reference materials were published in Applied Radiation and Isotopes.

Stability measurements were performed by measuring ²⁴¹Am by γ-spectrometry, for both reference materials. For short-term stability, extreme temperatures that could be reached during transport were tested, namely 4 °C and 40 °C. 20 °C was also tested, as a more usual temperature. Bottles of reference materials were placed at these temperatures for one week and measured afterwards. For long-term stability, two possible shelf temperatures, 4 °C and 20 °C, were tested for the liquid RM while for the solid, only 20 °C was tested. Several checks of the long-term stability of the reference materials were planned, and only the first one, after four months (June 2025) was performed as of September 2025. Additional measurements will be performed in October 2025 and February 2025.

The results were compared to the initial measurements performed for homogeneity testing (Figure 5).

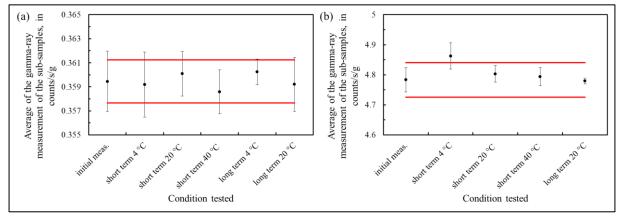


Figure 5. Comparison between the average of the measurements of the sub-samples for the initial measurement, for homogeneity (forty sub-samples), for the short-term stability samples kept at 4°C, 20 °C and 40 °C, and for the long-term stability samples kept at 4 °C and/or 20 °C (3 sub-samples each). (a) liquid reference material, (b) solid reference material. The bars represent the standard deviation among the sub-samples, and the red lines represent the uncertainty of the y-ray spectrometry measurement.

The homogeneity and stability measurements were used to calculate the contribution of those two parameters to the overall uncertainty of the measurands, following:

$$u_{RM}^2 = u_{char}^2 + u_{bb}^2 + u_{wb}^2 + u_{st}^2 + u_{lt}^2$$

Where

uncertainty of the measurement used to characterise one measurand of the reference material u_{char} :

uncertainty component coming from the difference between bottles of the reference material u_{bb} :

uncertainty component coming from the difference within bottles of the reference material u_{wb} :

uncertainty component coming from the difference in the reference material, after a short period u_{sts} : of time

uncertainty component coming from the difference in the reference material, after a long period u_{lt} :

The results are presented in Table 1. The liquid reference material seems to be more homogeneous and more stable than the solid one. This could be due to physical reasons: it is possible that it was harder to keep the radionuclides homogeneously distributed during the solidification process. The ²³⁸U and ²³⁹Pu solid reference material homogeneity tests were also performed using a less precise technique than the one used for the liquid reference material.















Newsletter 05 - Summer/Autumn 2025

Table 1. Uncertainty contributions coming from homogeneity and stability assessments, for both reference materials.

Reference material	Measurand	u_{bb}	u_{wb}	u_{sts}	u_{lt}	
Liquid	²³⁴ U, ²³⁵ U, ²³⁶ U and ²³⁸ U; U isotopic ratios	0.0%	0.2%	0.0%	0.0%	
	²³⁹ Pu and ²⁴⁰ Pu ; Pu isotopic ratios	0.0%	0.1%	0.0%	0.0%	
	²⁴¹ Am and ²³⁷ Np	0.0%	0.7%	0.0%	0.0%	
Solid	²³⁴ U, ²³⁵ U, ²³⁶ U and ²³⁸ U; U isotopic ratios	0.4%	3.8%	0.5%	0.0%	
	²³⁹ Pu and ²⁴⁰ Pu ; Pu isotopic ratios	0.9%	6.3%	0.5%	0.0%	
	²⁴¹ Am and ²³⁷ Np	0.7%	0.9%	0.5%	0.0%	

The assigned values for each measurands were determined either from direct measurements on the reference materials, and/or from measurements on the 'multi-RN spiking mixture' and/or from the measurement of the individual starting solutions.

The interlaboratory comparisons (ILC) involved each 13 partners. Ten partners sent results files for the liquid ILC, and nine sent results for the solid ILC. From two to seven results were obtained for each measurand. The results of each participating lab were compared to the assigned values by calculating the normalised deviation from the assigned value (e_p) , and the zeta score (ζ_p) , calculated as follows:

$$e_p = \frac{A_p - A_{RM}}{A_{RM}}$$

$$\zeta_p = \frac{A_p - A_{RM}}{\sqrt{u_p^2 + u_{RM}^2}}$$

The results for ²⁴¹Am for the solid RM are given here as an example.

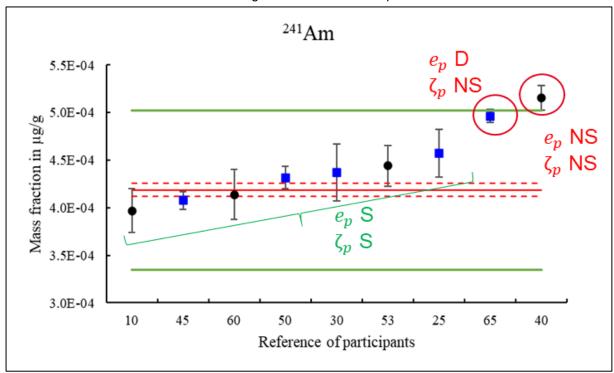


Figure 6. Results of the participants for the inter laboratory comparison based on the solid reference

The Deliverable D6 presents the results of the participants for the measurement of the mass fractions of 234 U, 235 U, 236 U, 237 Np, 238 U, 239 Pu, 240 Pu, 241 Am, the mass activity of 239 Pu + 240 Pu, the total U content and the 234 U/ 238 U, 235 U/ 238 U, 236 U/ 238 U and 240 Pu/ 239 Pu isotope ratios. Those results were put in perspective with the type of measurement (radiometric, mass spectrometry), and the sample preparation procedures.

Page: 10/15







JRP EPM 21GRD09 MetroPOEM Coordinator: Dirk Arnold (PTB)



Newsletter 05 – Summer/Autumn 2025

JRP EPM 21GRD09 MetroPOEM Coordinator: Dirk Arnold (PTB) E-mail: dirk.arnold@ptb.de

In summary, two reference materials were prepared, their homogeneity and stability were assessed using mass spectrometry and y-ray spectrometry, and the mass fraction of each radionuclide in the materials was measured. The content of both reference materials corresponded to slightly contaminated liquid and solid environmental samples. The final uncertainties for the measurands ranged from 0.3 % to 6.3 %, which was close to the requirements chosen at the beginning of the project.

The interlaboratory comparison allowed participating labs to compare their measurements both to the assigned values, and to other labs.

The reference materials produced are available for distribution to the community.

Development of SI traceable certified reference material Work Package 4:

The certified reference material, UME MetroPOEM CRM, was produced in accordance with the requirements of ISO 17034:2016 standard. The sampling of raw material was conducted as part of the regular monitoring research cruise AT 020, starting from Bremerhaven, Germany between 15-26 May 2023 covering the entire German EEZ as shown on the map in Figure 1. The equipment for ultra-trace water analysis for trace metals were prepared in accordance with GEOTRACES procedures and internally approved and validated protocols for trace analysis in seawater. The material was filtered through 0.2 µm membrane filters directly into precleaned carboys, acidified with HNO₃. After preliminary analysis at Hereon laboratories, material was transferred to TÜBİTAK UME and processed further. Considering the original and the target mass fractions of the elements the material was spiked by using NIST SRM 3100 series standards to obtain concentrations to be measured by analytical laboratories. All the materials in contact with the material and used in processing were cleaned by using sub-boiled HNO₃ and/or HCl and dried in ISO 6 clean chemistry laboratory (CCL) at TÜBİTAK UME. The cleanness of the bottles were checked by analysing the rinse solutions kept in the bottles for several days. After filling the homogenised material into the 250 mL polypropylene bottles (Figure 2) in the CCL, the material was sterilised by 25 kGy γ -radiation by 60 Co source. The total number of units obtained from the material is 470 and all of which was kept at 4 °C which was defined as the reference temperature for the material. Between unit homogeneity, long term stability (for 1 year at 21 °C) and short-term stability (3 weeks at 45 °C) were assessed in accordance with ISO 33405:2024 standard by 21GRD09 MetroPOEM Project partners. The random stratified sampling was used for selecting the units for tests. A combined measurement plan was conducted for homogeneity and stability with isochronous measurement design. The characterisation studies were conducted by an interlaboratory study where, in addition to the project partners, several expert organisations were also involved in isotope ratio measurements. All the assessments are based on the isotope ratio measurements conducted by using multi collector ICP-MS instruments except Ni for which mass fraction values determined by ICP-MS-MS were used for the assessment homogeneity and stability; thus only informative value is to be given in the certificate. The measurements methods employed matrix separation with suitable separation techniques. In addition to measurements carried out by MC-ICP-MS, single collector ICP-MS-MS measurements were also performed in the characterisation study. However, since the uncertainty obtained by later system is significantly higher, their results were not included in the certification. Uncertainties of the characterisation measurements were estimated in compliance with the Guide to the Expression of Uncertainty in Measurement (GUM). The uncertainties on the certified values are calculated according to the following equation (Eq. 1) which include uncertainties arising from possible between bottle heterogeneity u_{bb} , during transportation to the user laboratory premises u_{sts} , during its storage at user laboratory u_{lts} and the characterisation measurements u_{char} . The expended uncertainty with k factor corresponding to 95% confidence level were found to be less than 0.3% for majority of the isotopic ratio values. The highest uncertainty above 1% is found on $n(^{234}\text{U})/n(^{238}\text{U})$ The metrological traceability of the certified values to SI System of Units were ensured by use metrological traceable certified reference materials by the participating laboratories. The certified reference material is intended to be used for calibration of the instruments or quality control purposes in laboratories performing isotope ratio measurements.

$$U_{CRM} = k\sqrt{u_{char}^2 + u_{bb}^2 + u_{sts}^2 + u_{lts}^2}$$
 Eq. 1



















Newsletter 05 - Summer/Autumn 2025

Table 2. The mass fractions of the elements certified in UME MetroPOEM CRM and corresponding measurands

Elements	Mass fraction µg kg ⁻¹	Measurand	
B [†]	5400	n(11B)/n(10B)	
Li [†]	150	n(⁶ Li)/n(⁷ Li)	
Cd [‡]	12.5	n(¹¹⁴ Cd)/n(¹¹¹ Cd) n(¹¹⁴ Cd)/n(¹¹⁰ Cd)	
Ni ^{‡,}	13.4	n(60Ni)/n(58Ni)	
Pb [‡]	7.3	n(²⁰⁴ Pb)/n(²⁰⁶ Pb) n(²⁰⁷ Pb)/n(²⁰⁶ Pb) n(²⁰⁸ Pb)/n(²⁰⁶ Pb)	
U†	2.3	n(²³⁴ U)/n(²³⁸ U) n(²³⁵ U)/n(²³⁸ U)	



Figure 7. The stable elements in seawater certified reference material

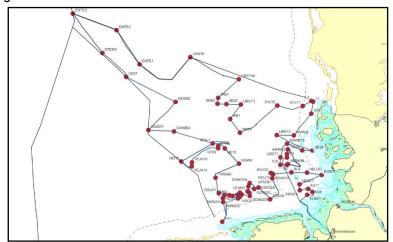


Figure 8. Route of the sampling cruise AT020

Page: 12/15

















JRP EPM 21GRD09 MetroPOEM **Coordinator: Dirk Arnold (PTB)**

[†] Determined by seaFast ICP-MS-MS by single laboratory

[‡] Determined by ID-ICP-MS by single laboratory

^{*} The isotope ration is given as informative value



Newsletter 05 - Summer/Autumn 2025

Work Package 5: Creating impact

The project Stakeholder Committee comprised 23 members from 14 countries, of which 11 are European. The total number of stakeholders was 55 members (83 persons) from 20 countries, of which 16 are European and 3 international organisations.

Presentations were made at various conferences and other events, and included of 9 invited oral presentations, 14 Oral presentations and 9 posters. These presentations are available at the project website

Peer reviewed papers

Analytical and Bioanalytical Chemistry

Flierl et al An advancement of the gravimetric isotope mixture method rendering the knowledge of the spike purity superfluous. https://doi.org/10.1007/s00216-024-05465-9

Analytical Chemistry

Lancaster et al $\delta(180/160)$ and $\delta(170/160)$ determinations in water using inductively coupled plasma-tandem mass spectrometry. https://doi.org/10.1021/acs.analchem.5c02607

Applied Radiation and Isotopes

- Lourenço et al Production of radioactive traceable reference materials for measuring radioactive pollutants in the environment. https://doi.org/10.1016/j.apradiso.2025.112204
- Arnold et al Progress achieved in EURAMET project 21GRD09 MetroPOEM: Metrology for the of environmental harmonisation of measurements pollutants Europe. in https://doi.org/10.1016/j.apradiso.2025.112182

Environmental Science and Technology

Time-Integrated Analysis of Sr, Ba, and 87Sr/86Sr in Freshwater using DGT Wagner et al Passive Samplers†.

European Physical Journal

Arnold et al Progress achieved in EURAMET project 21GRD09 MetroPOEM: Metrology for the harmonisation of measurements of environmental pollutants Europe. https://doi.org/10.1051/epjconf/202532311004

Journal of Analytical Atomic Spectroscopy

- Isobaric interference removal for selected radionuclides using nitrous oxide and Lancaster et al ammonia with inductively coupled plasma tandem mass spectrometry[†].
- Investigating the potential of methyl fluoride cell gas for radionuclide Russell et al interference removal and measurement using ICP-MS/MS[†].

Journal of Geophysical Research: Oceans

Pérez-Tribouillier, et al The Role of the St. Anna Trough in Atlantic Water Transport Into the Arctic Ocean: A Novel Radiogenic Isotope Assessment Using Iodine, Uranium, and Neodymium. https://doi.org/10.1029/2024JC022050

Nuclear Instruments and Methods in Physics Research Section B

Christl et al Results from the Zurich sea water (ZSW) intercomparison study for U- and Pu-isotopes. https://doi.org/10.1016/j.nimb.2025.165750

A training course was held as part of the NKS-B RadWorkshop 2024, (9-13 September 2024), at the DTU Risø Campus in Roskilde, Denmark, attended by 104 participants from nearly 60 organisations in 20 countries to 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.

Training videos, prepared by Montanuniversität Leoben, with input from other project partners during the course of the project, are available for viewing at the project website:

https://www.npl.co.uk/euramet/metropoem

Page: 13/15

















JRP EPM 21GRD09 MetroPOEM Coordinator: Dirk Arnold (PTB)

[†] DOIs for these papers will be available shortly



Newsletter 05 - Summer/Autumn 2025

Professional workshops were held at:

- **BIPM** Mass spectrometry workshop
- 32nd Symposium of the Radiation Protection Association of Serbia and Montenegro
- 10th International Conference on Nuclear and Radiochemistry
- 24th Winter plasma conference (M30) Workshops

Trade press

A trade press article, 'L'environnement sous surveillance : les prouesses de la spectrométrie de masse', by Valérie Lourenço (CEA), Lucille Chambon (CEA), Soumya Gupta (CEA), Hélène Isnard (CEA), Béatrice Lalère (LNE), Johanna Noireaux (LNE) is due to appear shortly in Actualité Chimique.

Standards committees

Project consortia members contributed to these standards and committees:

AFNOR: T90A and T90Q

BIPM: CCQM (IAWG), CCQM (IRWG), CCRI(II) and CCRI(II)-MS-TG

BSI: RMI/1

CEN: TC 230, TC 264 and TC 444

CIAAW **IUPAC:**

EURAMET: TC-IR and TC-MC

ISO: REMCO, TC 85 and TC 147

Social media

The project maintained a presence on LinkedIn and ResearchGate (unavailable after 30 September)

Stakeholder workshops

In addition to the third stakeholder workshop (described above), the first stakeholder workshop was held at the project start-up meeting and the second at the M18 project meeting.



















JRP EPM 21GRD09 MetroPOEM Coordinator: Dirk Arnold (PTB)

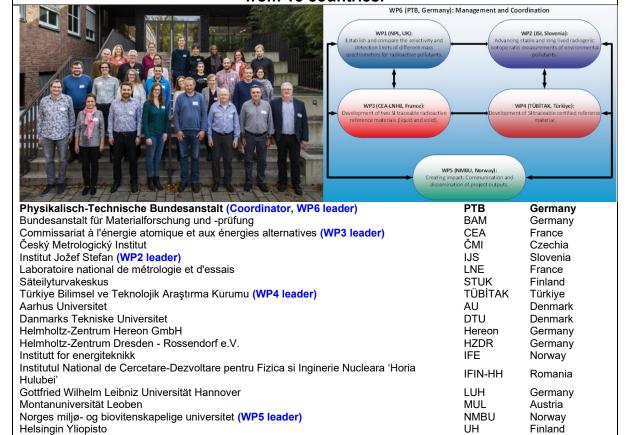
E-mail: dirk.arnold@ptb.de

Page: 14/15

Newsletter 05 – Summer/Autumn 2025

JRP EPM 21GRD09 MetroPOEM Coordinator: Dirk Arnold (PTB) E-mail: dirk.arnold@ptb.de

MetroPOEM, coordinated by the Physikalisch-Technische Bundesanstalt of Germany, was delivered by a consortium of 24 partners and 3 collaborators from 15 countries.



Institut za nuklearne nauke Vinča Institut od nacionalnog značaja za Republiku Srbiju, VINS Serbia Univerzitet u Beogradu UGOT Göteborgs universitet Sweden Universität Wien UNIVIE Austria Eidgenössische Technische Hochschule Zürich **ETHZ** Switzerland LGC Limited LGC NPL Management Limited (WP1 leader) NPL Collaborators Triskem Triskem International Fizinių ir technologijos mokslų centras

United Kingdom United Kingdom France **FTMC** Lithuania 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

MetroPOEM outputs are available from the project website, which will be maintained beyond

the end of the project

https://www.researchgate.net/profile/Metro-Poem Research gate:

The project 21GRD09 MetroPOEM has received funding from the European Partnership on Metrology, co-financed from the European Union's Horizon Europe Research and Innovation Programme and by the Participating States.

European Partnership on Metrology Funder Name:

10.13039/100019599 **Funder ID:** Grant number: 21GRD09 MetroPOEM







Spiez Laboratory

















