Metrology for the harmonisation of measurements of environmental pollutants in Europe Newsletter 03 - Summer 2024

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# News from 21GRD09 MetroPOEM

The second progress meeting (M18) was held at Laboratoire National de Métrologie et d'Essais in Paris, France on 19th-21st March 2024 and was attended by 36 participants in person and a further 16 on-line participants, including 14 from the stakeholder community.



Participants at the M18 meeting

# Project management board meeting

Eighteen out of the total of 23 members of the Project Management Board participated (12 in person, 6 online) in the meeting. Among others, the major decision was: 'All members (online and present) agreed by an unanimous vote to proceed with all activities of the project as planned in the JRP protocol and not to amend the proposed work in any activity. The due days of delayed activities will be adjusted in a way that all deliverables will be realised in the remaining 18 months.'

# Project progress meeting

Thirty-six persons from the project consortium (22 in person, 14 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:

- Materials for measurement have been prepared and although delayed, shipping is expected imminently. Other activities, including data collection formats are on schedule. Colleagues from MUL described an update on sample preparation.
- Method development, optimization of separations and evaluating isotope separation are all ongoing and on track. Short presentations of work were given by BAM, Hereon, LGC, MUL, PTB and TÜBİTAK.























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- WP3: Delays in material provision have impacted the work in WP3, but the plan has been amended to keep delays to a minimum; other activities are ongoing. Progress on the preparation of the solid reference material was noted, and the inactive material will be available soon.
- The water samples from the North Sea have been processed and preliminary measurements started. The techniques used to ensure that external contamination of the water samples were described and a table of responsibilities for particular measurements has been made.

Analyte	Responsible Partner	Measurement Technique
Boron	BAM	Isotope ratio
Lithium	BAM	Isotope ratio
Cadmium	JSI	Isotope ratio
Chromium	JSI	Isotope ratio
Nickel	MUL	Isotope ratio
Antimony	TÜBİTAK	IDMS
Lead	HEREON	Isotope ratio
Uranium	PTB	Isotope ratio

# Stakeholder meeting

The meeting was chaired by Paola Fisicaro, Division Head of Chemistry and Biology at France's Laboratoire national de métrologie et d'essais. The stakeholder participants were from AGES (AT), Agilent (UK), ARPA Lombardia (IT), BfS (DE), DSA (NO), Elemental Scientific Instruments (FR), IRSN (FR) as well as CCRI(II) and the ICRM (both international).

Progress in all the project work packages was reported, and the interactions with similar activities were briefly discussed.

### Next meeting

The next project management board, project progress and stakeholder meetings will be held in Risø 13th-15th November 2024.

Please contact Jixin Qiao, DTU, Denmark for registration forms, meeting details and information on accomodation at: Jixin Qiao <jiqi@dtu.dk>

# Science Feature - Preparation of samples for an inter-laboratory comparison exercise

This has been carried out by NPL (United Kingdom)

Mass spectrometry is increasingly applied to measurement of medium and long-lived radionuclides to support end users in environmental radioactivity, nuclear decommissioning, nuclear forensics and paleoclimate applications. This technique offers a rapid measurement alternative to some decay counting techniques and it also expands the number of radionuclides that are measurable.

As the popularity of mass spectrometry techniques for radionuclide measurement increases, it is critical that this is supported by underpinning standards. The aim of Work Package 1 is to provide a series of standards to test the capabilities of different mass spectrometric techniques for low-level radionuclide detection. This will inform end users of the relative strengths and limitations of different mass spectrometric techniques, as well as a comparison with decay counting techniques.

A series of individual and mixed standards of key radionuclides (Strontium-90, Uranium and plutonium isotopes, Americium-241 and Neptunium-237) have been prepared at the National Physical Laboratory, UK. These radionuclides were identified during formulation of the project, and a survey was completed by a number of laboratories on the activity levels of interest and their measurement capabilities. Multistage dilutions were made from standardised starting materials to very low activity levels in a dedicated source preparation facility. The dilution levels were checked at every stage for every individual and mixed radionuclide using inductively coupled plasma mass spectrometry (ICP-MS/MS, Figure). The impurity levels of the starting materials and final materials were also quantified for both stable and radioactive impurities using ICP-MS/MS and radiometric techniques, and the stability of the samples prepared are being measured at regular intervals by ICP-MS/MS for the duration of the project.



















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The dispatch team in the Nuclear Metrology Group at NPL are organising the distribution of samples to a number of analytical laboratories. Some of these labs have experience in measurement of radioactivity by mass spectrometry, and some for which this is relatively new regarding specific instrument techniques or mass spectrometry generally.

PTB have developed a reporting tool that allows the analyst to enter a range of details about the measurements made. These will be used to produce the interlaboratory comparison report. The final results will be combined with outputs from Work Package 3 to produce a Good Practice Guide on the use of mass spectrometry for radioactivity measurement.



Figure 1: Mass spectrometry facility at the National Physical Laboratory



















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# Short reports from the technical work packages.

### WP1: NPL

All standards for Work Package 1 have been prepared and are being distributed to the participating laboratories. There have been some delays with completing and approving the paperwork for each laboratory. Measurements will take place over the next few months and the interlaboratory comparison report will be completed by November 2024.

#### WP2: IJS

Currently, the performance of different mass spectrometric techniques (MC-ICP-MS, sector field ICP-MS, quadrupole ICP-MS, ICP-MS/MS) and existing calibration approaches are tested. For some elements, the first evaluations and comparison data, in terms of accuracy and precision of measurement results, are already available (Fig. 2) and show good agreement between measurement techniques for Pb isotope ratios. When applying reaction gases in ICP-MS/MS the precision should be improved. In this case, some additional optimisation is still required.

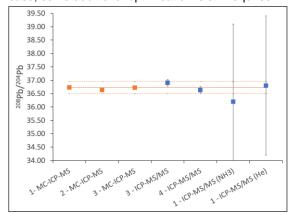


Figure 2: Comparison of Pb isotope ratios obtained by different mass spectrometers for NIST SRM 981.

Further, partners are working on the development of new techniques for the separation of selected elements from the sea matrix by testing different resins (Table 1) and automatic systems for preconcentration and/or matrix removal PrepFAST and SeaFAST.

Partner Lab	Isotopic system	Resin
Hereon	Pb	DGA
	В	sublimation
TÜBİTAK	Pb	Pb Resin Triskem
	В	"Amberlite IRA 743 AG 1-X8"
ВАМ	Li	AG 50W-X12
	В	"AG 50W-X8 Amberlite IRA 743"
	В	"AG 50W-X8 Microsublimation"
	Pb	Pb Resin Triskem
	Cd	AG 1-X8
AU	Pb	Anion exchange BioRad AG-1 X-8
	U	Anion exchange BioRad AG-1 X-8
JSI	Cd	BioRad AG 1-X8
		BioRad MP-1M
	Cr	BioRad AG 1-X8
IFE	В	Amberlite IRA 743
	Li	AG 50W-X12
MUL	Pb	Sr spec or DGA resin (Eichrom)
	Ni	Ni-Spec and others
LGC	Ni	Chelex 100 chelating resin

Table 1: Selection of resins for matrix removal for selected elements.

#### WP3: **CEA**

laboratories have completed measurements on the raw seawater either by mass spectrometry or by radiometric techniques. The intercomparison on the liquid RM is delayed for several reasons (knock on effects on radionuclide solution availability from WP1 and paperwork to cross European borders) but should start during the summer 2024. The intercomparison on the solid RM is still scheduled for October 2024. For this latter. CEA has successfully conducted small scale synthesis experiment of the solid RM and a real scale experiment which allowed to produce about 10 kg of inactive solid RM with at least a 3-fold decrease of the moisture uptake of the resulting material. Twenty 100-g samples of the inactive material were prepared among which 8 were sent to participants to test their dissolution procedures (Figures 3 and 4).



Figure 3: Picture of the batch of 20 samples of inactive solid reference material produced.



















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Figure 4: Image of the solid material sent (powder sieved to 250 µm)

### WP4: TÜBİTAK

The certified seawater reference material being developed to validate analytical methods, support proficiency testing, and ensure quality control in future monitoring is the main goal of the work package. To date, 250 litres of seawater have been collected from the North Sea and sent to TÜBİTAK and CMI laboratories. After cleaning procedures were applied to the entire processing equipment and bottles, 120 litres of the material were spiked with specific elements, homogenized, filtered, and bottled, resulting in 470 bottles. These bottles were gammairradiated and stored at +4 °C. Long-term stability testing was initiated in August 2023 for a year. The methods being developed in WP2 will be used in characterization work.

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### Management and dissemination.

### WP5: NMBU

Since the last newsletter, the project publishable summary and DCE plan have been updated. Project partners have made presentations at two IAEA Technical meetings, the Deutschen Physikalischen Gesellschaft meeting and the EURAMET TC IR meeting. There was a strong presence from the project at the recent Nordic Conference won Plasma spectrometry with two posters presented.

DTU will deliver a training course for the project as part of the upcoming RadWorkshop 2024 to be held at DTU in September:

https://www.conferencemanager\_dk/radworkshop20 24/workshop-programme

Additionally there will be a project presence at the 10th International Conference on Nuclear and Radiochemistry (NRC-10) in Brighton, UK during August:

https://www.rsc.org/events/detail/38385/10thinternational-conference-on-nuclear-andradiochemistry-nrc10

# WP6: PTB

The project successfully completed the mid-term review by EURAMET in April 2024 and the section of the report specific to MetroPOEM will be available on the project website. The main recommendations were:

- Maximise engagement with end users,
- Organise webinars and workshops (note: already part of the project), and
- Ensure the targets for open access and high impact peer reviewed publications are met.

The regular M18 reporting documents were sent to EURAMET in May 2024 and await approval.

A new project partner has joined MetroPOEM:

University of Vienna (UNIVIE)

UNIVIE will bring additional mass spectrometry skills and experience to the project.



















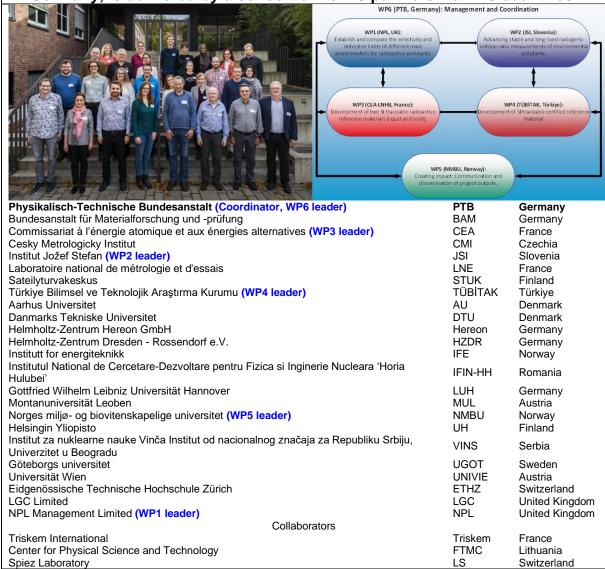


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MetroPOEM, coordinated by the Physikalisch-Technische Bundesanstalt of Germany, is delivered by a consortium of 23 partners from 13 countries.



# **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

https://www.linkedin.com/in/metropoem-project-308762251/ Linkedin:

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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