Metrology for the harmonisation of measurements of environmental pollutants in Europe Newletter 1 – Spring 2023 JRP EPM 21GRD09 MetroPOEM Coordinator: Dirk Arnold (PTB) E-mail: dirk.arnold@ptb.de

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# MetroPOEM, coordinated by the Physikalisch-Technische Bundesanstalt of Germany, is delivered by a consortium of 22 partners from 13 countries.



Physikalisch-Technische Bundesanstalt (Coordinator, WP6 leader)	РТВ	Germany
Bundesanstalt für Materialforschung und -prüfung	BAM	Germany
Commissariat à l'énergie atomique et aux énergies alternatives (WP3 leader)	CEA	France
Cesky Metrologicky Institut	CMI	Czechia
Institut Jožef Stefan (WP2 leader)	JSI	Slovenia
Laboratoire national de métrologie et d'essais	LNE	France
Sateilyturvakeskus	STUK	Finland
Türkiye Bilimsel ve Teknolojik Araştırma Kurumu <b>(WP4 leader)</b>	TÜBİTAK	Türkiye
Aarhus Universitet	AU	Denmark
Danmarks Tekniske Universitet	DTU	Denmark
Helmholtz-Zentrum Hereon GmbH	Hereon	Germany
Helmholtz-Zentrum Dresden - Rossendorf e. V.	HZDR	Germany
Institutt for energiteknikk	IFE	Norway
Institutul National de Cercetare-Dezvoltare pentru Fizica si Inginerie Nucleara 'Horia Hulubei'	IFIN-HH	Romania
Gottfried Wilhelm Leibniz Universität Hannover	LUH	Germany
Montanuniversität Leoben	MUL	Austria
Norges miljø- og biovitenskapelige universitet (WP5 leader)	NMBU	Norway
Helsingin Yliopisto	UH	Finland
Institut za nuklearne nauke Vinča Institut od nacionalnog značaja za Republiku Srbiju, Univerzitet u Beogradu	VINS	Serbia
Eidgenössische Technische Hochschule Zürich	ETHZ	Switzerland
LGC Limited	LGC	United Kingdom
NPL Management Limited (WP1 leader)	NPL	United Kingdom



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# Metrology for the harmonisation of measurements of environmental pollutants in Europe

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### Overview

The European Green Deal's ambition for zero pollution requires the development of highly sensitive techniques to detect ultra-low amounts of pollutants. This requirement will be delivered via strategies described by the two European Metrology Network (EMN) on Pollution Monitoring (PolMo) and the EMN on Radiation Protection, supporting the Basic Safety Standards directive. Implicit in these strategies is a strong need to improve data quality for monitoring and reporting pollution in the air, water, and soil. In addition, the lack of suitable traceability chains and appropriate quality control compromises the comparability and robustness of measurements.

To detect radioactive isotopes and stable polluting elements in the environment, fast, sensitive, and inexpensive analytical procedures are needed. Mass spectrometry is a key method for determination of non-radioactive polluting elements and is of increasing importance for long-lived radionuclides. Despite the increasing application of single collector ICP-MS, this potential cannot be fully realised unless techniques can be validated with traceable multi-element reference materials. However, multi-element certified reference materials are usually not available and single-element certified reference materials are limited to very few elements. Nevertheless, these reference materials are urgently needed to calibrate mass spectrometric measurements, due to mass bias effects occurring during the measurements in mass spectrometers.

#### Objectives

The overall aim of MetroPOEM is 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. The aims and objectives of MetroPOEM will be delivered through 4 technical work packages, supported by project and impact management activities:

- Establish and compare (inter-laboratory) the selectivity and detection limits of diverse types of mass spectrometers 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.
- Develop measurement methods for isotope ratios that are traceable to the SI by using multi-collector 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.
- 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 WP1, which will demonstrate the variations in parameters including detection limits, sample preparation, sample introduction methods, total procedural time, and uncertainty budgets.

- 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, to facilitate the calibration of single collector ICP-MS, instrument validation, as well as quality control.
- Facilitate the acceptance 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 and end users (e.g., environmental monitoring agencies).

### Project Outcomes

MetroPOEM will enable and harmonise measurement methods for the detection and characterisation, of both radioactive isotopes and stable polluting elements, in support of the EU Green Deal's aim toward a zero pollution, toxic-free environment. The new reference materials (RMs) developed in this project will address the ongoing need to produce suitable and relevant RMs that can validate measurement capabilities.

The scientific outcome of the proposed research will deliver validated and traceable analytical approaches for the analysis of the concentration of pollutants, as well as



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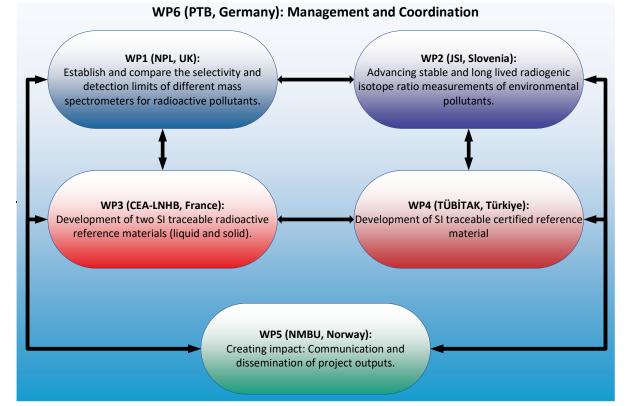
determining the source and monitoring any contamination of pollutants through isotope ratio measurements. This will close existing metrological gaps and will lead to a harmonisation of methods.

MetroPOEM will deliver an improved system of metrology and will establish an infrastructure that directly supports the application of EU regulations or EU directives. By implementing new traceability chains, different methods will be combined in the field of pollution monitoring, which will then lower the detection limits. This will result in better protection of the environment, provide new tools for complex studies in climate observation, support validated data collection. Additionally, accurate JRP EPM 21GRD09 MetroPOEM Coordinator: Dirk Arnold (PTB) E-mail: dirk.arnold@ptb.de

waste classification engenders public confidence and ensures inventories are correct for future infrastructure planning, such as the scale and design of pollutant remediation programmes.

The outputs from the project may be employed in several diverse fields including routine realtime monitoring, emergency response, geological dating, and climate change studies, and in other activities, such as nuclear forensics, decommissioning non-nuclear industries. The collaboration between European laboratories established in this project is expected to continue beyond the end of the project.

### Project work packages



#### WP1: Establish and compare the selectivity and detection limits of different mass spectrometers

The aim of this work package is to establish the capabilities of different mass spectrometry designs using radionuclide standard solutions. The focus will be on relative instrument performance with respect to current measurement challenges around detection limits and interference removal capabilities and establishing detection limits in relation to environmental regulations, and in comparison, to decay counting techniques. The work

package will focus on multiple radioactive pollutants (<sup>237</sup>Np, <sup>234,235,236,238</sup>U, <sup>239,240</sup>Pu, <sup>241</sup>Am and possibly <sup>226</sup>Ra and <sup>90</sup>Sr), all of which are known to be measurable by mass spectrometry, at activities relevant to regulatory limits. The range of radionuclides chosen are well suited for comparison based on the range of half-lives, the presence of multiple spectral interferences that must be overcome to ensure accurate measurement, and precise isotopic ratio measurements (<sup>239</sup>Pu/<sup>240</sup>Pu, <sup>236</sup>U/<sup>238</sup>U) for source identification. Mass spectrometry is increasingly used for measuring a growing

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number of radionuclides, but this is not currently supported by underpinning standard solutions for instrument calibration and method validation.

# WP2: Advancing stable and long-lived radiogenic isotope ratio measurements of environmental pollutants

The aim of this work package is to develop new and improved generic methods for stable and radioactive isotope lona-lived ratio measurements by mass spectrometry with uncertainties that allow resolving natural mass dependent isotope fractionation. The environmentally relevant elements selected as key indicators for the development and optimisation of measurement procedures include Li, B, Cr, Cd, Ni, Sb, Pb, and U.

## WP3: Development of radioactive reference materials

The aim of this work package is to produce two reference materials (RMs), respectively liquid and solid, containing radioactive pollutants (<sup>237</sup>Np, <sup>234,235,236,238</sup>U, <sup>239,240</sup>Pu, <sup>241</sup>Am and possibly <sup>226</sup>Ra and <sup>90</sup>Sr) addressing end users and stakeholders needs. These RMs will be used in two inter-laboratory comparisons employing techniques used in WP1 to meet objective one and will demonstrate the variations in parameters including detection limits, sample preparation requirements,

sample introduction methods, total procedural time, and uncertainty budgets.

Existing radioactive RMs and CRMs for environmental samples that may be measured by mass spectrometry are very limited and their characterisation does not always include relevant parameters such as isotopic ratios. This information would help characterise the mass bias related to the matrix effect. WP3 will focus on the development of a liquid RM and a solid RM which may also be used for QC measurements.

### WP4: Development of SI traceable certified reference material

The aim of this work package is to produce a certified reference material (CRM) for inorganic environmental pollutants which will be designed according to the needs of the end users performing environmental analysis and monitoring. The production and certification of the material will be carried out in accordance with EN ISO 17034 standard (General requirements for the competence of reference material producers) requirements.

#### WP5: Creating impact

This task will deal with the communication and dissemination of project outputs via the stakeholder committee and project website. Publications will be through the scientific literature, other publications and presentations at appropriate events, workshops, and training material.

### **Project information**

The overall deliverables and dissemination routs are tabulated 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: <u>metropoem@nmbu.no</u>



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### **Project deliverables**

N°	Objective	Description	Туре	Partners	Delivery date
D1	1	Inter-laboratory comparison report describing low-level radioactive pollutants (e.g., U, Np, Pu, Am) by different types of mass spectrometers	Inter- laboratory comparison report	<b>NPL</b> , CEA, DTU, ETHZ, HZDR, NMBU, PTB	M24 Sep-24
D2	1	Good Practice Guide on the use of mass spectrometry for low-level radioactive pollutants detection	Good Practice Guide	NPL, BAM, CEA, DTU, ETHZ, HZDR, IFIN-HH, IJS, LGC, LUH, MUL, NMBU, PTB, UH	M36 Sep-25
D3	2	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 on more commonly available techniques by providing suitable operating procedures focussing on stable polluting elements	Report	<b>JSI</b> , AU, BAM, DTU, Hereon, IFE, IFIN- HH, LGC, LNE, LUH, MUL, NPL, PTB, TÜBİTAK	M28 Jan-25
D4	2	Good Practice Guide on sample processing, treatment, uncertainty budgets, and if feasible, the quantification of the so-called mass bias	Good Practice Guide	LGC, AU, BAM, DTU, Hereon, IFIN- HH, IJS, LNE, MUL, NPL, PTB, TÜBİTAK	M36 Sep-25
D5	3	Document describing two radioactive reference materials, developed and shipped to participants for their radioactive content characterisation	Document	CEA	M24 Sep-24
D6	3	Inter-laboratory comparison report, describing detection limits, sample preparation requirements, sample introduction methods, total procedural time, and uncertainty budgets	Inter- laboratory comparison report	CEA, ČMI, DTU, ETHZ, HZDR, IFIN- HH, IJS, LUH, NMBU, PTB, STUK, UH, VINS	M36 Sep-25
D7	4	Report describing the development of one aqueous certified reference material that is certified for the same stable polluting elements with lowest possible uncertainties	Report	TÜBİTAK, AU, BAM, DTU, Hereon, IFE, IJS, LGC, LNE, MUL, PTB	M36 Sep-25
D8	n/a	Evidence of contributions to the Stakeholder Committee, professional workshops, discussion group, training courses, e-training and to new or improved international standards and recommendations with a specific focus on ISO/TC 085/SC 02/WG 17, ISO/TC 147/SC 3, CEN/TC 230, CEN/TC 264, CEN/TC 444. Examples of early uptake of project outputs by end users. Updated dissemination, communication and exploitation plan.	Reporting documents	<b>NMBU</b> , all participants	M36 Sep-25
D9	n/a	Delivery of all technical and financial reporting documents as required by EURAMET	Reporting documents	PTB, all participants	M36 + 60 days Nov-25



### Metric POEM Metrology for the harmonisation of measurements of environmental pollutants in Europe Newletter 1 – Spring 2023

### **Project impact pathways**

SPECIFIC NEEDS	EXPECTED RESULTS	DCE MEASURES
What are the specific needs that triggered this project?	What do you expect to generate by the end of the project?	What dissemination, communication and exploitation measures will you apply to the results?
European Green Deal's ambition for zero pollution requires the detection of ultra-low amounts of pollutants and determination their isotope ratios. Many pollutants in the environment exist at extremely low concentrations, which are below contemporary detection limits. Extremely sensitive mass spectrometry techniques are now within the grasp of many routine monitoring laboratories. However, these novel methods are often not traceable nor validated, particularly for the elements / pollutants concerned, and hence need to be compared and harmonised on the European scale.	New measurement techniques for the characterisation and detection of radioactive and stable polluting elements that provide harmonised data, traceable to national and international standards. Closure of the traceability gap arising from lack of definitive knowledge of how isotope fractionation effects impact measurement quality and traceability. Reference materials that support the measurement of very low levels of elemental pollutants.	Dissemination of the project outcomes via peer reviewed publications, conference presentations, user workshops, engagement in measurement networks and provision of project data to suitable databases. Development and publication of a GPG that draws together best practice for environmental measurements of elemental pollutants. <i>Communication</i> of the project outcomes via a range of dissemination routes to the general public, academia, governmental agencies and industry. <i>Exploitation</i> will be realised by the uptake of project outcomes, such as traceable isotope ratio measurements and improved analytical performance by pollutant monitoring laboratories.
TARGET GROUPS	OUTCOMES	IMPACTS
Who will use or further up-take the results of the project? Who will benefit from the results of the project?	What change do you expect to see after successful dissemination and exploitation of project results to the target group(s)?	What are the expected wider scientific, economic and societal effects of the project contributing to the expected impacts outlined in the work programme and call scope?
Scientific community: Measurement and calibration laboratories. Stakeholder and end users: Instrument and material manufacturers: Including Agilent, Nu Instruments, Shimadzu UK, TrisKem.	Uptake of the scientific work of the project through more specific measurements, lower achievable detection limits, traceable measurements below the mBq/µBq and pg/fg activity and mass ranges.	Scientific: General improvement in monitoring capabilities. Economic: Better pollution control through attribution and targeted remediation. Support for European Green Deal's ambition for zero pollution. Societal: Reduction in pollutant impact on the environment from a range of industrial sectors.

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