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NUSIMEP-8: Uranium and plutonium isotope amount ratios in low-level synthetic nitrate solution

Inter-laboratory comparison, Report to participants

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Abstract

The Additional Protocol (AP) authorizes safeguards authorities to verify the absence of undeclared nuclear activities in all parts of a state's nuclear fuel cycle as well as any other location where nuclear material is or may be present. As a part of the Additional Protocol, environmental sampling has become an important tool for the detection of non-declared nuclear activities. In environmental sampling, swipe samples are collected for bulk and particle analysis. Considering the potential consequences of the analyses, these measurements need to be subjected to a rigorous quality management system. The Nuclear Signatures Inter-laboratory Measurement Evaluation Programme (NUSIMEP) was established in 1996 to support the growing need to trace and measure the isotopic abundances of elements characteristic for the nuclear fuel cycle present in trace amounts in the environment. NUSIMEP-8 focused on measurements of low-level uranium and plutonium in synthetic nitrate solution aiming to support EURATOM safeguards (DG ENER), the IAEA Network of Analytical Laboratories (NWAL) for bulk analysis of environmental samples and laboratories in the field. The NUSIMEP-8 solution was prepared from mixed oxide fuel dissolved in nitric acid with addition of natural uranium and diluted to an environmental level. Participating laboratories in NUSIMEP-8 received one sample solution with undisclosed values of $n(^{238}Pu)/n(^{239}Pu)$, $n(^{240}Pu)/n(^{239}Pu)$, $n(^{241}Pu)/n(^{239}Pu)$, $n(^{242}Pu)/n(^{239}Pu)$ and $n(^{234}U)/n(^{238}U)$, $n(^{235}U)/n(^{238}U)$, $n(^{236}U)/n(^{238}U)$ amount ratios. Those isotope amount ratios were measured by participating laboratories using their routine analytical procedures. Measurement of the major isotope ratios $n(^{235}U)/n(^{238}U)$ and $n(^{240}Pu)/n(^{239}Pu)$ were obligatory; measurement of the minor isotope ratios were optional. 25 laboratories registered for NUSIMEP-8, three withdrew the registration while one laboratory encountered problems with the shipment of the sample. Finally, 19 participants have reported measurement results using different analytical techniques, among those 10 NWAL laboratories. Two participants did not report their results due to technical problems. The participant measurement results have been evaluated against the certified reference values by means of z-scores and zeta-scores in compliance with ISO 13528:2005. The NUSIMEP-8 results were overall satisfactory and in compliance with the IAEA Measurement Quality Goals for the analysis of bulk environmental samples. This report presents the NUSIMEP-8 participant results; including the evaluation of the questionnaire. In addition feedback from the measurement communities in nuclear safeguards, nuclear security and environmental sciences was collected in view of identifying future needs for NUSIMEP inter-laboratory comparisons.

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NUSIMEP

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Summary

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The Nuclear Signatures Inter-laboratory Measurement Evaluation Programme (NUSIMEP) was established in 1996 to support the growing need to trace and measure the isotopic abundances of elements characteristic for the nuclear fuel cycle present in trace amounts in the environment. NUSIMEP-8 focused on measurements of low-level uranium and plutonium in synthetic nitrate solution aiming to support EURATOM safeguards (DG ENER), the IAEA Network of Analytical Laboratories (NWAL) for bulk analysis of environmental samples and laboratories in the field.

The NUSIMEP-8 solution was prepared from mixed oxide fuel dissolved in nitric acid with addition of natural uranium and diluted to an environmental level. Participating laboratories in NUSIMEP-8 received one sample solution with undisclosed values of $n(^{238}\mathrm{Pu})/n(^{239}\mathrm{Pu})$, $n(^{240}\mathrm{Pu})/n(^{239}\mathrm{Pu})$, $n(^{241}\mathrm{Pu})/n(^{239}\mathrm{Pu})$, $n(^{242}\mathrm{Pu})/n(^{239}\mathrm{Pu})$ and $n(^{234}\mathrm{U})/n(^{238}\mathrm{U})$, $n(^{235}\mathrm{U})/n(^{238}\mathrm{U})$, $n(^{236}\mathrm{U})/n(^{238}\mathrm{U})$ amount ratios. Those isotope amount ratios were measured by participating laboratories using their routine analytical procedures. Measurement of the major isotope ratios $n(^{235}\mathrm{U})/n(^{238}\mathrm{U})$ and $n(^{240}\mathrm{Pu})/n(^{239}\mathrm{Pu})$ were obligatory; measurement of the minor isotope ratios were optional. 25 laboratories registered for NUSIMEP-8, three withdrew the registration while one laboratory encountered problems with the shipment of the sample. Finally, 19 participants have reported measurement results using different analytical techniques, among those 10 NWAL laboratories. Two participants did not report their results due to technical problems. The participant measurement results have been evaluated against the certified reference values by means of z-scores and zeta-scores in compliance with ISO 13528:2005. The NUSIMEP-8 results were overall satisfactory and in compliance with the IAEA Measurement Quality Goals for the analysis of bulk environmental samples. This report presents the NUSIMEP-8 participant results; including the evaluation of the questionnaire. In addition feedback from the measurement communities in nuclear safeguards, nuclear security and environmental sciences was collected in view of identifying future needs for NUSIMEP inter-laboratory comparisons.

1. Introduction

Nuclear safeguards arrangements exist on international level under the protocols of the International Atomic Energy Agency (IAEA) [1] on European Union level under the EURATOM Treaty [2] and on regional levels. The INFCIRC/540 also referred to as the Additional Protocol (AP), moved the focus from exclusively accounting for known quantities of fissile material towards a more qualitative system that is able to provide a comprehensive picture of a state's nuclear activities [3]. Through unannounced inspections and nuclear material balances, safeguards inspectors are able to verify that no nuclear material is diverted from its intended peaceful use. As part of the Additional Protocol, environmental sampling has become an important tool for the detection of non-declared nuclear activities. Analysis of environmental samples is carried out to detect the (unavoidable) traces in the environment originating from technological activities. One extensively developed technique in environmental sampling makes the use of cotton or cellulose swipes to wipe surfaces inside and around a nuclear facility. Bulk analysis of these swipe samples represents an average concentration and isotope abundance of uranium and plutonium in the whole sample [4]. The swipe sample is first decomposed, followed by a chemical separation and finally measured by mass spectrometric technique using Thermal Ionization Mass Spectrometry (TIMS) or Inductive Coupled Plasma Mass Spectrometry (ICP-MS). This method is able to detect uranium and plutonium concentrations in the picogram range.

The IRMM Nuclear Signatures Inter-laboratory Measurement Evaluation Programme (NUSIMEP) is an external quality control programme organised by the Institute for Reference Materials and Measurements of the Joint Research Centre (JRC-IRMM). NUSIMEP was established in 1996 to support the growing need to detect and measure the isotopic abundances of elements characteristic for the nuclear fuel cycle present in trace amounts in the environment. Such measurements are required for safeguards applications as well as for the implementation of the Treaty on the Non-Proliferation of Nuclear Weapons (NPT) [3]. Measurements of the isotopic ratios of the uranium and plutonium in small amounts, such as typically found in environmental samples, are required for nuclear safeguards, for the control of environmental contamination and for the detection of nuclear proliferation.

Laboratories participating in NUSIMEP are requested to measure the parameters specified using their standard analytical procedures and report measurement results with associated uncertainties to JRC-IRMM. The reported measurement results are compared with independent external certified reference values with demonstrated traceability and uncertainty, as evaluated according to international guidelines. Laboratory performance evaluation is done according to the respective ISO standard on performance evaluation in proficiency testing by inter-laboratory comparisons [5].

Laboratories analysing environmental samples are invited to participate in these external NUSIMEP quality control exercises to demonstrate and assess their ability to carry out accurate measurements in particular on trace amounts of uranium and plutonium. Through this and similar programmes, the degree of equivalence of measurements of individual laboratories can be ascertained.

Several NUSIMEP inter-laboratory comparisons of measurements of uranium isotopic ratios were organised previously: for example NUSIMEP-2, uranium isotopic abundances in dry uranium nitrate samples; NUSIMEP-3, uranium isotopic abundances in saline media, NUSIMEP-4, uranium isotopic abundances in simulated urine and NUSIMEP-5 uranium, plutonium and caesium isotopic ratios in saline medium. Reports of the previous NUSIMEP inter-laboratory comparisons can be found on the IRMM website [6].

The organisation of the inter-laboratory comparison follows the standard procedures of the Inter-laboratory Measurement Evaluation Programmes IMEP, REIMEP, and NUSIMEP of the Institute for Reference Materials and Measurements (IRMM) of the Joint Research Centre, a Directorate-General of the European Commission. This programme is accredited according to ISO/IEC 17043:2010 [7].

2. Scope and aim

Measurements of the isotopic ratios of the elements uranium and plutonium in small amounts, such as typically found in environmental samples, are required for the control of environmental contamination and for the detection of nuclear proliferation. NUSIMEP-8 aims at laboratories carrying out bulk analysis in these various application fields. Particular emphasis was given to participation of the IAEA network of analytical laboratories for environmental sampling (NWAL) [8]. Participation of the NWAL laboratories in this NUSIMEP inter-laboratory comparison was formally recommended by the IAEA at the IAEA Technical Meeting on Bulk Analysis of Environmental Samples for Safeguards.

The JRC-IRMM and JRC-ITU joined efforts to provide, in the frame of REIMEP-17 that was organised in parallel for nuclear plant operators and nuclear material laboratories, also 'low-level' samples suitable for a NUSIMEP inter-laboratory comparison in support to environmental laboratories and the IAEA-NWAL [9]. The measurands in NUSIMEP-8 were $n(^{238}Pu)/n(^{239}Pu)$, $n(^{240}Pu)/n(^{239}Pu)$, $n(^{241}Pu)/n(^{239}Pu)$, $n(^{242}Pu)/n(^{239}Pu)$ and $n(^{234}U)/n(^{238}U)$, $n(^{235}U)/n(^{238}U)$, $n(^{236}U)/n(^{238}U)$ amount ratios. The NUSIMEP-8 sample was prepared in 1 $mol \cdot L^{-1}$ nitric solution containing about 10 $ng \cdot g^{-1}$ U and 0.11 $ng \cdot g^{-1}$ Pu in a screw cap ampoule. The accompanying letter with the participation key, the guidelines on result reporting, the sample receipt form, and a checklist was also delivered together with the sample. Measurement of the major ratios $n(^{235}U)/n(^{238}U)$ and $n(^{240}Pu)/n(^{239}Pu)$ were obligatory; measurement of the minor ratios were optional, but it was recommended to report also the minor ratios.

3. Time frame

NUSIMEP-8 was announced for participation on April 1, 2012. The deadline for registration was May 15, 2012. The confirmation of registration was sent to the participants and subsequently the samples were dispatched between June 2012 and May 2013 from JRC- ITU Karlsruhe. The originally reporting deadline from April 1, 2013 had to be extended to July 1, 2013. The extension of the deadline was necessary because the coordination of NUSIMEP-8 was aligned time-wise with REIMEP-17 on synthetic input solution, which involved nuclear transport [9]. Due to difficulties with the transport there was a delay in shipping the NUSIMEP-8 samples to two participants. The homogeneity and short term stability studies were finalised at JRC-IRMM in July 2013. The certified reference values were sent to the participants on October 2, 2013.

4. Test material

4.1. Preparation of the solution

The NUSIMEP-8 solution was prepared by gravimetric dilution of REIMEP-17 mother solution. The mother solution was prepared by dissolution of a mixed oxide fuel in nitric acid (p.a. Merck, Darmstadt, Germany) and with addition of natural uranium aiming at concentration of uranium and plutonium of about 200 $mg \cdot g^{-1}$ and 2 $mg \cdot g^{-1}$, respectively. This solution was further diluted to a final concentration of uranium and plutonium of about 10 $ng \cdot g^{-1}$ and 0.11 $ng \cdot g^{-1}$, respectively. After the homogenization, the solution was dispensed into screw cap ampoules with a peristaltic pump. 70 ampoules of NUSIMEP-8 were prepared, each containing about 10 ml sample solution of 1 $mol \cdot L^{-1}$ nitric acid (supra pure). The dispensing and the sealed ampoules of NUSIMEP-8 are shown in Figure 1.

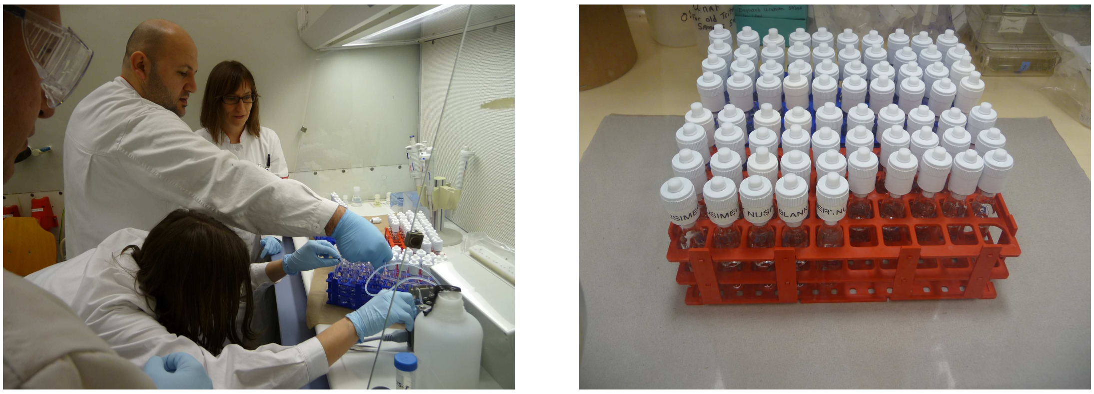

Figure 1: Dispensing of a NUSIMEP-8 sample solution with a peristaltic pump on a clean bench (left) and the sealed ampoules of NUSIMEP-8 (right) at JRC-ITU-Karlsruhe.

4.2. NUSIMEP-8 value assignment

The reference values in NUSIMEP-8 were established by Thermal Ionisation Mass Spectrometry (TIMS) [9, 10, 11]. The NUSIMEP-8 sample was prepared by a three-step gravimetric dilution of the REIMEP-17 mother solution, and the verification measurements of the REIMEP-17 mother solution carried out at JRC-ITU confirmed the reference values within measurement uncertainties established at JRC-IRMM. The external verification of the isotope amount ratios in the two fractions of REIMEP-17 allowed a different approach for the value assignment for the uranium and plutonium isotope amount ratios in NUSIMEP-8. Therefore, the design of the study was such that the value assignment for REIMEP-17 and NUSIMEP-8 were combined [9]. Assuming, it is very unlikely that isotope fractionation occurred during the gravimetric dilution of the higher concentrated fractions of REIMEP-17 to the lower concentrated fraction of NUSIMEP-8, the value assignment for the major and minor isotope amount ratios in NUSIMEP-8 was done by TIMS on the samples of the fraction REIMEP-17A for a higher accuracy and only verified as far as possible with respect to the homogeneity and stability assessment for NUSIMEP-8, see also paragraph 4.3 and 4.4.

The target relative standard uncertainty for method repeatability in NUSIMEP-8 was about < 0.5% for the major (e.g. most abundant) isotope amount ratios and 10% for the minor isotope ratios. This goal was met in NUSIMEP-8 for all the minor plutonium isotope amount ratios, measured with a relative standard uncertainty for method repeatability ranging from 0.3% - 2%. The relative method repeatability for the major plutonium ratio $n(^{240}Pu)/n(^{239}Pu)$ was 0.2%. Due to an analytical error during the chemical separation step of the NUSIMEP-8 samples the uranium fraction could not be assessed for homogeneity and stability. Additional measurements could not be performed because of limited human resources and time constraints, nevertheless the ILC organisers were confident in homogeneity and stability of the uranium isotope ratios in NUSIMEP-8 and were considered fit for purpose. In addition, participants in NUSIMEP-8, who stated to be experts in the field, could reproduce the NUSIMEP-8 reference values for the major and minor uranium isotope amount ratios, which was an additional external confirmation for the ILC organisers that no contamination or fractionation occurred during sample preparation. Admittedly, one drawback of this approach is that the relative expanded uncertainty of the NUSIMEP-8 reference value for $n(^{236}U)/n(^{238}U)$ is larger than the respective IAEA Measurement Quality Goals for the analysis of bulk environmental samples [12].

4.3. Homogeneity

As JRC-IRMM is not only an accredited ILC provider but at the same time an accredited producer of similar reference materials of nuclear reference materials, the homogeneity assessment was done in compliance with ISO Guide 35:2006 [13] and the IUPAC International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories [14].The minimum number of units for the homogeneity study , $N_{min} = max\left(10, \sqrt[3]{N_{produced}}\right)$, was chosen according to recommendations given in ISO Guide 35:2006 paragraph 7.4.1 [13]. According to the design of the study the homogeneity was assessed via measurement of isotope amount ratios by TIMS in five randomly selected ampoules of NUSIMEP-8. The results from the measurements of the plutonium isotope ratios of the five NUSIMEP-8 samples were evaluated by a one-way analysis of variance (ANOVA) [15, 16 , 17]. This allows the separation of the method variation (s_{wb}) from the experimental averages over the replicates measured in one bottle to obtain estimation for the real variation between bottles (s_{bb}), with u^*_{bb} being the lower limit to the between bottle variance which depends on the mean squares between bottles, the number of replicate measurements per bottle and the degrees of freedom of the mean squares within bottles. It can be understood as the “detection limit” of the homogeneity study. The uncertainty of homogeneity is consequently estimated as s_{bb} or in case of $s_{bb} < u^*_{bb}$ as u^*_{bb}. This approach, applying single factor ANOVA as described in [15, 16, 17] is compliant with ISO Guide 35:2006, the IUPAC Harmonized Protocol and was found to be comparable to tests to determine whether an ILC material is sufficiently homogeneous for its purpose as described in ISO 13528 [5]. Essentially, these tests compare the unit heterogeneity with the standard deviation for proficiency assessment. Assessment criterion for a homogeneity check is s_{bb} (or u^*_{bb}) ≤ 0.3 $\hat{\sigma}$. The results of the homogeneity assessment in NUSIMEP-8 are listed in Annex 1.

The standard deviation for proficiency assessment was set in compliance with the IAEA Measurement Quality Goals for the analysis of bulk environmental samples. Laboratories to qualify for the Network of Analytical Laboratories (NWAL) for environmental sampling have to demonstrate that they meet the requirement set in the respective IAEA procedure. The IAEA Measurement Quality Goals are expressed for $n(^{240}Pu)/n(^{239}Pu)$, $n(^{241}Pu)/n(^{239}Pu)$, $n(^{242}Pu)/n(^{239}Pu)$ and $n(^{234}U)/n(^{238}U)$, $n(^{235}U)/n(^{238}U)$, $n(^{236}U)/n(^{238}U)$ at specific values of the ratios as relative expanded uncertainties [12]. Furthermore, there is no IAEA Measurement Quality Goal for $n(^{238}Pu)/n(^{239}Pu)$. Therefore, $\hat{\sigma}$ for $n(^{238}Pu)/n(^{239}Pu)$ was set as for the other plutonium isotope amount ratios. The variation between units (s_{bb}) for all plutonium amount ratios in NUSIMEP-8 is listed in Table 1. The tests indicate that the NUSIMEP-8 test material is sufficiently homogeneous for the plutonium amount ratios.

4.4. Stability

The 'short term' stability assessment was carried out one year after the preparation of the NUSIMEP-8 samples with the aim of confirming the reference values. This was necessary because NUSIMEP-8 was organised in parallel with REIMEP-17 and depending on licenses and shipment requirements for different countries the shipment of the samples was performed over a rather large timespan, see also paragraph 3. The samples selected for short term stability assessment were stored at room temperature and measured by TIMS at JRC-IRMM. Methods to assess whether an ILC material is sufficiently stable for its purpose are described in ISO 13528 [5]. Essentially, these tests compare the general averages of the measurand obtained in the homogeneity check (x_s) with those obtained in the stability check (y_s). The absolute difference of these averages is again compared to the standard deviation for proficiency assessment $\hat{\sigma}$. The assessment criterion for a stability check in ISO 13528 is $|x_s-y_s| \leq 0.3\hat{\sigma}$. As can be seen from Table 1 the criterion was met for the stability of all the plutonium isotope amount ratios in NUSIMEP-8. The ILC organisers assumed the samples to be fit for purpose with respect to the homogeneity of uranium isotope ratios as described in paragraph 4.2, since the samples were prepared by gravimetric dilution of the REIMEP-17 mother solution and since the plutonium isotope amount ratios were found to be homogeneous. The results from the homogeneity and stability assessment are summarized in Table 1.

Table 1: Homogeneity and stability tests for NUSIMEP-8 according to ISO 13528 [5]

NUSIMEP-8	*Relative* s_{bb}	standard deviation for proficiency assessment $\hat{\sigma}$	Homogeneity check $s_{bb} \leq 0.3\hat{\sigma}$	Stability check $|x_s-y_s| \leq 0.3\hat{\sigma}$
$n(^{234}U)/n(^{238}U)$*	-	$0.05X_{ref}$	-	-
$n(^{235}U)/n(^{238}U)$*	-	$0.005X_{ref}$	-	-
$n(^{236}U)/n(^{238}U)$*	-	$0.05X_{ref}$	-	-
$n(^{238}Pu)/n(^{239}Pu)$	1.15%	$0.05X_{ref}$	YES	YES
$n(^{240}Pu)/n(^{239}Pu)$	0.14%	$0.05X_{ref}$	YES	YES
$n(^{241}Pu)/n(^{239}Pu)$	0.35%	$0.05X_{ref}$	YES	YES
$n(^{242}Pu)/n(^{239}Pu)$	0.42%	$0.05X_{ref}$	YES	YES

(*) Due to an analytical error during the chemical separation step of the NUSIMEP-8 samples the uranium fraction could not be assessed for homogeneity and stability. Nevertheless the ILC organisers were confident that the NUSIMEP-8 samples are fit for purpose as described in paragraph 4.2

4.5. Distribution

The ILC samples were dispatched to the participants from JRC-ITU Karlsruhe between June 2012 and May 2013 via regular carrier service since the total activity of the sample was below 1000 Bq. Each participant received a package with one ampoule of NUSIMEP-8 sample solution; the accompanying letter with the participation key, the guidelines on result reporting, and a form to confirm the receipt of the package. As mentioned before, for logistic reasons NUSIMEP-8 samples were shipped together with REIMEP-17 samples to participants taking part in both ILCs.

5. Participant invitation, registration and information

Participation of the NWAL laboratories in this NUSIMEP-8 inter-laboratory comparison was formally recommended by the IAEA. Furthermore, NUSIMEP-8 was announced in relevant conferences and meetings. Invitations were sent to the NWAL laboratories and other participants who expressed their interest in participation via e-mail. Measurement of the major ratios $n(^{235}U)/n(^{238}U)$ and $n(^{240}Pu)/n(^{239}Pu)$ were obligatory; measurement of the minor ratios $n(^{234}U)/n(^{238}U)$, $n(^{236}U)/n(^{238}U)$ and $n(^{238}Pu)/n(^{239}Pu)$, $n(^{241}Pu)/n(^{239}Pu)$, $n(^{242}Pu)/n(^{239}Pu)$ were optional. Participants were asked to follow their routine procedures.

Participants were informed that their measurement results would be evaluated against the certified reference values and that full confidentiality would be guaranteed with respect to the link between measurement results and the participants' identity. The call for participation was also announced on the IRMM website (Annex 2). The confirmation of registration was sent to those participants who had registered (Annex 3). The Accompanying letter with the instructions on measurands and measurements were sent to the participants together with a sample (Annex 4). The letter also contained the individual code to access via the respective website the result reporting and the related questionnaire pages (Annex 5). After sample receipt, the participants had to return the signed 'Confirmation of sample receipt' form (Annex 6). In addition, a guide to help the participants with the online result reporting tool was also provided.

Table 2 lists the number of registered participants per country.

Table 2: Number of registered participants per country

Country	Number of participants
Australia	2
Austria	1
Brazil	1
China	1
Finland	1
France	1
Germany	1
Greece	1
Hungary	1
Italy	1
Republic of Korea	1
Sweden	2
Switzerland	1
The Netherlands	1
United Kingdom	3
United States	2

6. NUSIMEP-8 reference values

Table 3 lists the NUSIMEP-8 reference values X_{ref} and their associated expanded uncertainties U_{ref} (k = 2).

Table 3: NUSIMEP-8: uranium and plutonium isotope amount ratios in low-level synthetic nitrate solution reference values

NUSIMEP-8	Isotope amount ratio	
	Certified value [1)] [mol/mol]	Uncertainty [2)] [mol/mol]
$n(^{234}U)/n(^{238}U)$	0.0000657	0.0000015
$n(^{235}U)/n(^{238}U)$	0.0068092	0.0000057
$n(^{236}U)/n(^{238}U)$	0.0000029	0.0000015
$n(^{238}Pu)/n(^{239}Pu)$	0.042596	0.000042
$n(^{240}Pu)/n(^{239}Pu)$	0.478692	0.000055
$n(^{241}Pu)/n(^{239}Pu)$	0.12573	0.00023
$n(^{242}Pu)/n(^{239}Pu)$	0.137468	0.000038

1) The reference date for the certified values is March 1, 2013.
2) The certified uncertainty is the expanded uncertainty with a coverage factor $k = 2$ corresponding to a level of confidence of about 95 % estimated in accordance with ISO/IEC Guide 98-3, Guide to the Expression of Uncertainty in Measurement (GUM:1995), ISO, 2008

7. Reported results

7.1. General observations

19 participants submitted results in NUSIMEP-8 and completed the associated questionnaire, among those 10 NWAL laboratories. The laboratories were asked to apply their routine measurement procedure and to report their results for the isotope amount ratios with uncertainties and the respective coverage factors. Measurement of the major ratios, $n(^{235}U)/n(^{238}U)$ and $n(^{240}Pu)/n(^{239}Pu)$, were obligatory; measurement of the minor ratios $n(^{234}U)/n(^{238}U)$, $n(^{236}U)/n(^{238}U)$, $n(^{238}Pu)/n(^{239}Pu)$, $n(^{241}Pu)/n(^{239}Pu)$ and $n(^{242}Pu)/n(^{239}Pu)$, were optional. It was highly recommended to report also the minor ratios. Participants from the same institute applying more than one analytical method had to register separately. Two laboratories could not report results due to technical problems. All laboratories that submitted results reported values for the $n(^{235}U)/n(^{238}U)$ isotope amount ratios, 17 laboratories reported values for the $n(^{240}Pu)/n(^{239}Pu)$ isotope amount ratio, 18 participants reported values for the minor ratio $n(^{234}U)/n(^{238}U)$ and 14 for the $n(^{236}U)/n(^{238}U)$. 10 participants also reported the values for the $n(^{238}Pu)/n(^{239}Pu)$ amount ratios, 12 participants for the $n(^{241}Pu)/n(^{239}Pu)$ amount ratio and 15 participants $n(^{242}Pu)/n(^{239}Pu)$ amount ratio. All results in NUSIMEP-8 are listed as reported by the participants. Table 4 shows the reported results per participant.

Table 4: Reported results per participant

Country	$n(^{234}U)/n(^{238}U)$	$\mathbf{n(^{235}U)/n(^{238}U)}$	$n(^{236}U)/n(^{238}U)$
Australia	✓	✓	✓
Australia	✓	✓	
Austria	✓	✓	✓
Brazil	✓	✓	✓
China	✓	✓	✓
Finland	✓	✓	
France	✓	✓	✓
Germany	✓	✓	✓
Greece	✓	✓	
Hungary	✓	✓	
Italy	Technical problem		
Korea, Rep. of	✓	✓	✓
Netherlands		✓	
Sweden	✓	✓	✓
Sweden	✓	✓	✓
Switzerland	✓	✓	✓
United Kingdom	✓	✓	✓
United Kingdom	✓	✓	✓
United Kingdom	✓	✓	✓
United States	✓	✓	✓
United States	Technical problem		

Country	$n(^{238}Pu)/n(^{239}Pu)$	$\mathbf{n(^{240}Pu)/n(^{239}Pu)}$	$n(^{241}Pu)/n(^{239}Pu)$	$n(^{242}Pu)/n(^{239}Pu)$
Australia		✓		✓
Australia	✓	✓		✓
Austria	✓	✓	✓	✓
Brazil		✓	✓	✓
China		✓		✓
Finland				
France		✓	✓	✓
Germany		✓		
Greece	✓	✓	✓	✓
Hungary		✓	✓	
Italy	Technical problem			
Korea, Rep. of	✓	✓	✓	✓
Netherlands	✓	✓	✓	✓
Sweden	✓	✓	✓	✓
Sweden	✓	✓	✓	✓
Switzerland		✓	✓	✓
United Kingdom	✓	✓	✓	✓
United Kingdom	✓	✓	✓	✓
United Kingdom	✓	✓	✓	✓
United States				
United States	Technical problem			

7.2. Measurement results

Annexes 7-13 list the individual measurement results and display overview graphs.

8. Scoring of results

8.1. The scores and their settings

Individual laboratory performance is expressed in terms of z and zeta scores in accordance with ISO 13528 [5].

$$z = \frac{x_{lab} - X_{ref}}{\hat{\sigma}} \quad \text{and} \quad zeta = \frac{x_{lab} - X_{ref}}{\sqrt{u_{ref}^2 + u_{lab}^2}}$$

Where

x_{lab} is the measurement result reported by a participant

X_{ref} is the certified reference value (assigned value)

u_{ref} is the standard uncertainty of the reference value

u_{lab} is the standard uncertainty reported by a participant

$\hat{\sigma}$ is the standard deviation for proficiency assessment

Both scores can be interpreted as: satisfactory result for $|score| \le 2$, questionable result for $2 < |score| \le 3$ and unsatisfactory result for $|score| > 3$.

z score

The NUSIMEP-8 *z* score indicates whether a laboratory is able to perform the measurement in accordance with the IAEA Safeguards Analytical Services Measurement Quality Goals for the analysis of bulk environmental samples (IAEA-SGAS-QG) [12]. The NUSIMEP-8 standard deviations for proficiency assessment $\hat{\sigma}$ are listed in Table 5.

Table 5: NUSIMEP-8 standard deviations for proficiency assessment

NUSIMEP-8	standard deviation for proficiency assessment $\hat{\sigma}$ in compliance with IAEA-SGAS-QG [12]
$n(^{234}U)/n(^{238}U)$	$0.05X_{ref}$
$n(^{235}U)/n(^{238}U)$	$0.005X_{ref}$
$n(^{236}U)/n(^{238}U)$	$0.05X_{ref}$
$n(^{240}Pu)/n(^{239}Pu)$	$0.05X_{ref}$
$n(^{241}Pu)/n(^{239}Pu)$	$0.05X_{ref}$
$n(^{242}Pu)/n(^{239}Pu)$	$0.05X_{ref}$

zeta score

The zeta score provides an indication of whether the estimate of uncertainty is consistent with the laboratory's deviation from the reference value [5]. It is calculated only for those results that were accompanied by an uncertainty statement. The interpretation is similar to the interpretation of the z score. An unsatisfactory zeta score may be caused by an underestimated uncertainty or by a large deviation from the reference value. The standard uncertainty of the laboratory (u_{lab}) was calculated as follows: if an uncertainty was reported, it was divided by the coverage factor k. If no coverage factor was provided, the reported uncertainty was considered as the half-width of a rectangular distribution. The reported uncertainty was then divided by √3, in accordance with recommendations issued by Eurachem and CITAC [18, 24].

acceptable uncertainty

Since the IAEA-SGAS-QG are expressed as relative expanded uncertainty (95% confidence interval), a performance assessment criterion for minimum and maximum acceptable uncertainty to complete satisfactory scores that take reported measurement uncertainties into account was applied in NUSIMEP-8 [19, 20, 21, 22].

$$\text{for all } \|\text{zeta}\| \leq 2\text{; it is evaluated whether } 0 < u_{lab;rel} \leq \text{IAEA-SGAS-QG}$$

Where

$u_{lab;rel}$ is the relative standard uncertainty of the reported uncertainty by a participant

IAEA-SGAS-QG is the respective IAEA Quality Goal [12] expressed as relative combined standard uncertainty.

The interpretation is that for each satisfactory zeta score it was evaluated whether the relative reported standard uncertainty is within the respective IAEA-SGAS-QG. If this was the case then 'YES' was issued, otherwise 'NO'.

Furthermore, the IUPAC International Harmonised Protocol [14] suggests that participants can apply their own scoring settings and recalculate the scores if the purpose of their measurements is different.

8.2. Scoring the reported measurement results

A z score was calculated for all participants except for those who reported no value or an upper limit, "<" value. A zeta score was calculated for results that were accompanied by an uncertainty statement. Whether the uncertainty was acceptable or not was only evaluated for satisfactory zeta scores. Annexes 6-12 list the scores per measurand and participant in detail.

Table 6 summarises the scores per measurand under investigation. As there are no IAEA-SGAS-QG defined for the $n(^{238}\text{Pu})/n(^{239}\text{Pu})$, there were no z scores issued for this respective plutonium isotope amount ratio. The total number of participants in NUSIMEP-8 (with and without a score) is nineteen. It has to be kept in mind that participants can apply their own scoring settings and recalculate the scores if the purpose of their measurements is different [14]. It can be concluded that the majority of participants in NUSIMEP-8 in general performed well and in compliance with the respective IAEA-SG-QG, but for measurements of the $n(^{235}\text{U})/n(^{238}\text{U})$ amount ratio only less than half of the participants achieved satisfactory scores. This was partly due to the fact that the IAEA-SG-QG is more stringent for that specific ratio. As previously mentioned in paragraph 4.2 the drawback in NUSIMEP-8 is that the relative expanded uncertainty of the NUSIMEP-8 reference value for $n(^{236}\text{U})/n(^{238}\text{U})$ is larger than the respective IAEA Measurement Quality Goals for the analysis of bulk environmental samples [12]. This means that the uncertainty of the $n(^{236}\text{U})/n(^{238}\text{U})$ reference value is too large for the purpose of this ILC, which can easily be seen in Table 6 and Annex 8 by the increase of satisfactory zeta scores compared to the high number of unsatisfactory z scores. For the other isotope amount ratios 63% - 82% achieved satisfactory zeta scores, with even 100% of acceptable uncertainty results for $n(^{240}\text{Pu})/n(^{239}\text{Pu})$ and $n(^{242}\text{Pu})/n(^{239}\text{Pu})$.

Table 6: Overview of scores: S(atisfactory), Q(uestionable), U(nsatisfactory; n is the number of results for which a score was given.

NUSIMEP-8	z score				zeta score				acceptable uncertainty for $\|\text{zeta}\| \le 2$	z and zeta scores and uncertainty
	S	Q	U	n	S	Q	U	n	YES	S
$n(^{234}U)/n(^{238}U)$	75%	13%	13%	16	69%	6%	25%	16	82%	50%
$n(^{235}U)/n(^{238}U)$	41%	6%	53%	17	47%	12%	41%	17	50%	24%
$n(^{236}U)/n(^{238}U)$	18%	18%	64%	11	82%	9%	9%	11	56%	9%
$n(^{238}Pu)/n(^{239}Pu)$	-	-	-	-	63%	13%	25%	8	-	-
$n(^{240}Pu)/n(^{239}Pu)$	100%	-	-	15	87%	-	13%	15	100%	87%
$n(^{241}Pu)/n(^{239}Pu)$	82%	-	18%	11	64%	-	36%	11	71%	45%
$n(^{242}Pu)/n(^{239}Pu)$	85%	8%	8%	13	69%	-	31%	13	100%	69%

9. Further information extracted from the results

In addition to submission of the results, the participants were asked to answer questions related to the measurement protocols. All participants completed the questionnaire. Issues that may be relevant to the outcome of the inter-laboratory comparison are discussed in the following paragraphs.

9.1. Method of analysis

For the measurement of uranium isotope amount ratios, 10 participants applied Inductively Coupled Plasma Mass Spectrometry, 6 participants Thermal Ionisation Mass spectrometry (TIMS), 1 participant Accelerator Mass Spectrometry (AMS) and 2 participants alpha spectrometry. For the measurement of plutonium isotope amount ratios, 12 participants applied ICP-MS, 4 participants TIMS, 1 participant AMS and 2 participants used alpha spectrometry. One participant applied liquid scintillation counting (LSC) for determination of the $n(^{241}Pu)/n(^{239}Pu)$ isotope amount ratio.

9.2. A representative study

15 (79%) laboratories indicated that the NUSIMEP-8 sample was treated according to the same analytical procedure routinely used in their laboratory. 12 out of 19 participants reported that they are experienced in this type of measurement. 6 participants indicated that they analyse 11-50 samples per year, 7 participants analyse more than 100 samples per year. All of the laboratories are certified, accredited or authorised for this type of analysis.

The mission of the majority of laboratories participating in NUSIMEP-8 is to carry out measurements for fissile material control or safeguards and for environmental sciences. 10 laboratories indicated they are part of the Network of Analytical Laboratories (NWAL). Other laboratories are involved in research and development, one laboratory is from the clinical field. More than 50% of the laboratories routinely analyse soil and sediment samples, other analyse surface, sea or drinking water, urine samples and various biota samples. 6 laboratories reported that they analyse swipe samples. Some laboratories analyse specials samples such as molybdenum and nuclear waste, blood samples, faecal ash and others.

9.3. Quality system and use of standards

All laboratories except one reported that they are working according to a quality management system; either according to ISO 17025 and/or ISO 9000 series [23]. 17 out of 19 participants confirmed the participation in various inter-laboratory comparisons. The ILC schemes mentioned were NUSIMEP, REIMEP, Procorad, NPL, EQRAIN, ILCs organised by DOE and IAEA and others. All the participants, except one, routinely use certified reference materials for instrument calibration and for method validation. The certified reference materials used by the NUSIMEP-8 participants are given in Annex 13.

9.4. Determination of measurement uncertainty

All the participants except one stated that they routinely report uncertainties on measurements to their customers. The majority of the participants (16 out of 19) are familiar with the Guide for Quantifying Measurement Uncertainty (GUM) issued by the International Organization for Standardization (ISO, 2005) and/or EURACHEM/CITAC (2000) [18, 24] and applied those guides when estimating their measurement uncertainty in NUSIMEP-8. The other participants estimated their measurement uncertainty by standard deviation based on replicate measurements.

10. Feedback

One participant complained about the late arrival of the NUSIMEP-8 sample. This delay was due to the fact that the shipment to this participant was originally planned together with the shipment of the respective REIMEP-17 samples. Another participant could not measure the plutonium amount ratios due to delays in obtaining Pu CRMs from JRC-IRMM. One participant particularly expressed the usefulness of NUSIMEP inter-laboratory comparisons.

10.1. Outlook on future NUSIMEP ILCs

All the participants, except one, expressed interest in future NUSIMEP ILCs. Participants expressed that they would be interested in certified swipe sample test material for bulk analysis of uranium and plutonium. Some participants would be interested in samples containing uranium particles. Other participants mentioned nitric acid or water samples, urine and blood samples, forensic samples and others. The concentration range participants expressed were ng/g for uranium samples and fg/g or pg/g for plutonium samples. Among the elements, plutonium and uranium were mentioned; however there was also interest in thorium, americium, radium and other elements.

11. Conclusion

Environmental sample analysis is a powerful tool for the verification of the correctness and completeness of States' declarations and for attribution of intercepted materials, so that there is credible assurance of the non-diversion of nuclear material from declared activities and of the absence of undeclared nuclear activities. To this end bulk analyses of swipe samples taken by safeguards inspectors at nuclear facilities have become an integral part of the Additional Protocol.

Laboratories from the IAEA-NWAL for the bulk analysis of environmental swipe samples but also from the IAEA NWAL for nuclear material analysis successfully demonstrated their measurement capabilities via participation in NUSIMEP-8. The advantage of organising REIMEP-17 and NUSIMEP-8 in parallel was that laboratories with expertise in nuclear material analysis could with little additional analytical effort assess also their measurement performance for low-level plutonium and uranium isotope ratio measurements. Participation in NUSIMEP-8 of laboratories from the IAEA-NWAL for nuclear material analysis and of institutes whose mission is not necessarily environmental sample analysis was extremely useful and of mutual benefit to the participants and to the NUSIMEP-8 organisers.

It can be concluded that the participants in NUSIMEP-8 performed extremely well for the measurements of the plutonium amount ratios and the $n(^{234}U)/n(^{238}U)$ amount ratio. A larger spread of results for the $n(^{236}U)/n(^{238}U)$ amount ratio was to be expected due to the fact that ^{236}U is the least abundant isotope in the NUSIMEP-8 sample. It was somewhat surprising that only less than 50% of the participants could meet the IAEA Measurement Quality Goal for the major uranium ratio. But it has to be taken into account that this specific IAEA Measurement Quality Goal is 10 times more stringent than for all the other amount ratios. For some of the isotope ratios differences in the uncertainty estimates provided by laboratories were observed, even when using the same instrumental technique.

Acknowledgements

The authors would like to thank Frances Kehoe and Celia Venchiarutti for support in chemical separations and TIMS measurements; Marleen Peetermans, Sandra Vanhoovels for their administrative help at JRC-IRMM, and the colleagues from JRC-ITU Ramon Carlos-Marquez, Lorenza Emblico, Ana Sanchez-Hernandez, Martin Vargas-Zuniga, and Adrian Nicholl for the preparation of the solutions, the sealing of the ampoules as well as the packaging for transport. The mass spectrometric measurements for the initial stability and characterisation assessment have been performed by Joan Horta, Eckhard Dahms and Sylvain Millet in ITU. In addition we thank Corinne Brossard, Fernando Martinez, and Jacqueline Ribeiro for their cooperation throughout the project.

Abbreviations

AMS	Accelarator Mass Spectrometry
ANOVA	Analysis of Variance
AP	Additional Protocol
CETAMA	Commission d'Etablissement des Méthodes d'Analyse
CITAC	Co-operation for International Traceability in Analytical Chemistry
CRM	Certified Reference Material
DG ENER	Directorate General for Energy
ES	Environmental Sampling
ESARDA	European Safeguards Research and Development Association
EQRAIN	Evaluation de la Qualité des Résultats d'Analyse dans l'Industrie Nucléaire)
EU	European Union
EURATOM	European Atomic Energy Community
EURACHEM	A focus for Analytical Chemistry in Europe
GUM	Guide to the Expression of Uncertainty in Measurement
IAEA	International Atomic Energy Agency
ICP-MS	Inductively Coupled Plasma Mass Spectrometry
ILC	Interlaboratory Comparison
IMEP	Interlaboratory Measurement Evaluation Programme
JRC-IRMM	Institute for Reference Materials and Measurements, JRC, European Commission
ISO	International Organization for Standardization
JRC-ITU	Institute for Transuranium Elements, JRC, European Commission
IUPAC	International Union for Pure and Applied Chemistry
JRC	Joint Research Centre
LSC	Liquid Scintillation Counting
NBL	New Brunswick Laboratory
NPL	National Physical Laboratory
NPT	Treaty on the Non-Proliferation of Nuclear Weapons
NUSIMEP	Nuclear Signatures Interlaboratory Measurement Evaluation Programme
NWAL	Network of Analytical Laboratories
REIMEP	Regular European Interlaboratory Measurement Evaluation Programme
SGAS	Safeguards Analytical Services
SME	Safeguard Measurement Evaluation Programme
TIMS	Thermal Ionisation Mass Spectrometry

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Annexes

Annex 1: Results of the homogeneity assessment in NUSIMEP-8
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Annex 14: Summary of the infomartion given by the participants from the questionnaire

Annex 1: The results of the homogeneity and stability assessment in NUSIMEP-8

NUSIMEP-8	$n(^{238}Pu)/n(^{239}Pu)$			$n(^{240}Pu)/n(^{239}Pu)$		
Ampoule ID	Rep 1	Rep 2	Rep 3	Rep 1	Rep 2	Rep 3
8	0.042512	/	/	0.4764	/	/
16	0.042362	0.042457	0.042691	0.4766	0.4764	0.4783
24	0.043255	0.042855	/	0.4769	0.4762	/
48	0.042811	0.042493	0.045451	0.4769	0.4757	0.4749
56	0.043006	0.04276	/	0.4797	0.4772	/
mean	0.042968			0.4768		
$\hat{\sigma}_{rel}$ [%]	5.0			5.0		
0.3* $\hat{\sigma}_{rel}$ [%]	1.5			17		
$s_{bb,\ rel}$ [%]	MSB<MSW			0.14		
$s_{wb,\ rel}$ [%]	2.22			0.24		
$u_{bb,\ rel}$ [%]	1.15			0.12		
$s_{bb,\ rel}$ ($u_{bb,\ rel}$)< 0.3* $\hat{\sigma}_{rel}$	YES			YES		
$\lvert x_s - y_s \rvert$ [%].	1			4.2		
$\lvert x_s - y_s \rvert \leq 0.3\hat{\sigma}$.	YES			YES		

NUSIMEP-8	$n(^{241}Pu)/n(^{239}Pu)$			$n(^{242}Pu)/n(^{239}Pu)$		
Ampoule ID	Rep 1	Rep 2	Rep 3	Rep 1	Rep 2	Rep 3
8	0.12359	/	/	0.13864	/	/
16	0.12324	0.12316	0.12392	0.13826	0.13873	0.13894
24	0.12369	0.12307	/	0.13892	0.14168	/
48	0.12320	0.12265	0.12297	0.13827	0.13998	0.14077
56	0.12440	0.12409	/	0.1389	0.13808	/
mean	0.12345			0.13920		
$\hat{\sigma}_{rel}$ [%]	5.0			5.0		
0.3* $\hat{\sigma}_{rel}$ [%]	4.4			4.9		
$s_{bb,\ rel}$ [%]	0.35			0.12		
$s_{wb,\ rel}$ [%]	0.29			0.81		
$u_{bb,\ rel}$ [%]	0.15			0.42		
$s_{bb,\ rel}$ ($u_{bb,\ rel}$)< 0.3* $\hat{\sigma}_{rel}$	YES			YES		
$\lvert x_s - y_s \rvert$ [%].	0.7			4		
$\lvert x_s - y_s \rvert \leq 0.3\hat{\sigma}$.	YES			YES		

Annex 2: Invitation letter

Geel, 28 March 2012
JRC.D.2/YAr/Rbu/mt/ARES(2012) 12-045/361112

The IRMM Nuclear Signatures Interlaboratory Measurement Evaluation Programme

NUSIMEP-8: Interlaboratory Comparison on low-level uranium and plutonium in synthetic nitrate solution

NUSIMEP is an external quality control programme organised by IRMM with the object of providing materials for measurements of trace amounts of nuclear materials in environmental matrices. Measurements of the isotopic ratios of the elements uranium and plutonium in small amounts, such as typically found in environmental samples are required for nuclear safeguards, for the control of environmental contamination and for the detection of nuclear proliferation. Several NUSIMEP comparison campaigns of measurements of uranium isotopic ratios were organised previously: for example NUSIMEP-2, uranium isotopic abundances in dry uranium nitrate samples; NUSIMEP-3, uranium isotopic abundances in saline media; NUSIMEP-4, uranium isotopic abundances in a simulated urine; NUSIMEP-5 uranium, plutonium and caesium isotopic ratios in saline medium, NUSIMEP-6 and NUSIMEP-7 uranium isotope amount ratios in uranium particles.

http://irmm.jrc.ec.europa.eu/interlaboratory_comparisons/nusimep

We would like to announce the forthcoming NUSIMEP-8 interlaboratory comparison: "Uranium and plutonium isotope amount ratios in low-level synthetic nitrate solution" and invite laboratories to participate. Participating laboratories in NUSIMEP-8 receive one solution sample with undisclosed isotope amount ratio values $n(^{238}Pu)/n(^{239}Pu)$, $n(^{240}Pu)/n(^{239}Pu)$, $n(^{241}Pu)/n(^{239}Pu)$, $n(^{242}Pu)/n(^{239}Pu)$ and $n(^{234}U)/n(^{238}U)$, $n(^{235}U)/n(^{238}U)$, $n(^{236}U)/n(^{238}U)$. Those isotope amount ratios are to be measured by participating laboratories using their routine analytical procedures. Measurement of the major ratio $n(^{235}U)/n(^{238}U)$ and $n(^{240}Pu)/n(^{239}Pu)$ is obligatory; measurement of the minor ratios plutonium and uranium amount ratios are optional. The measurement results will be evaluated against the certified reference values. Full confidentiality is guaranteed with respect to the link between measurement results and the participants' identity.

The participation fee in NUSIMEP-8 is **€ 600**, which includes sample dispatch. "NUSIMEP-8" is an environmental like sample (activity <1000 Bq). The samples will be shipped from the Institute for Transuranium Elements (EC-JRC-Karlsruhe) to the participants using regular express carrier services. Due to the nature of this comparison only a limited number of samples are available. Samples will be allocated to participants in order of registration until the stock of NUSIMEP-8 samples is exhausted. Each participant has to request his/her import licence if needed in time to enable shipment.

We ask each participant to provide the following information:

1) contact person (e-mail address and telephone number)

2) contact person for licensing questions (*e.g.* import licence)

3) delivery address (not a PO Box, but a real address)

Retieseweg 111, 2440 Geel, Belgium
Tel.: +32-(0)14-571 623 • Fax: +32-(0)14-571 863
jrc-irmm-nusimep@ec.europa.eu • http://www.irmm.jrc.be

European Commission

NUSIMEP

Please register electronically for this interlaboratory comparison using the following link:

https://web.jrc.ec.europa.eu/ilcRegistrationWeb/registration/registration.do?selComparison=860

Once you have submitted your registration electronically, please follow the procedure indicated: a) print your registration form; b) sign it; and c) fax it to us. Your fax is the confirmation of your participation.

The deadline for registration is 30 April 2012. Samples will be sent to participants May-June 2012. The deadline for submission of results is 1 April 2013. The late result reporting deadline is due to the fact that the coordination of NUSIMEP-8 has to be aligned time-wise with REIMEP-17, which is organised in parallel.

Please do not hesitate to contact us in case you need more information.

Yours sincerely,

Renáta Buják
NUSIMEP-8 Co-ordinator

Yetunde Aregbe
IRMM Safeguards Co-ordinator

Retieseweg 111, 2440 Geel, Belgium
Tel.: +32-(0)14-571 623 • Fax: +32-(0)14-571 863
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Annex 3: Confirmation of registration

BUJAK Renata (JRC-GEEL)

From: BUJAK Renata (JRC-GEEL) on behalf of JRC IRMM NUSIMEP
Sent: 30 April 2012 11:11
To:
Subject: Successful registration to NUSIMEP-8

Dear

Thank you for registering to the NUSIMEP-8: low-level uranium, plutonium in synthetic nitrate solution interlaboratory comparison.

My colleagues from JRC-ITU Karlsruhe will contact you soon about the shipment of the sample.

Should you have further questions, please do not hesitate to contact me.

Kind regards,

Renata Bujak

NUSIMEP-8 co-ordinator

****

NUSIMEP
European Commission - Joint Research Centre
Institute for Reference Materials and Measurements (IRMM)
Retieseweg 111, B-2440 GEEL, BELGIUM
Tel: +32 14 571 623
Fax: +32 14 571 863
New-email: jrc-irmm-nusimep@ec.europa.eu
Web: http://irmm.jrc.ec.europa.eu; http://irmm.jrc.ec.europa.eu/catalogue

Annex 4: Accompanying letter

Geel, 13 June 2012
JRC.D.2/RB/mt/ARES(2012) 12-067/ 720934

«TITLE» «FIRSTNAME» «SURNAME»
«ORGANISATION»
«DEPARTMENT»
«ADDRESS»
«ADDRESS2»
«ADDRESS3»
«Address4»
«ZIP» «TOWN»
«COUNTRY»

NUSIMEP-8: Interlaboratory Comparison on low-level uranium and plutonium in synthetic nitrate solution

Dear «TITLE» «SURNAME»,

Thank you for your participation in NUSIMEP-8.
Together with this letter we are sending to you one ampoule of NUSIMEP-8 solution sample for analysis with undisclosed isotope amount ratios of uranium and plutonium as described in the NUSIMEP-8 announcement:

http://irmm.jrc.ec.europa.eu/interlaboratory_comparisons/nusimep/nusimep-8/Pages/index.aspx

Please check whether the ampoule remained intact during transport and then sign the "Confirmation of receipt" form and return it by email or fax to us (Fax: +32 14 571 863).

Participants in NUSIMEP are asked to apply the same measurement procedure as used in routine sample analysis of this kind.

The uranium and plutonium isotope amount ratios are to be measured. Measurement of the major ratios: $n(^{235}U)/n(^{238}U)$ and $n(^{240}Pu)/n(^{239}Pu)$ are obligatory; measurement of the minor ratios $n(^{234}U)/n(^{238}U)$, $n(^{236}U)/n(^{238}U)$ and $n(^{238}Pu)/n(^{239}Pu)$, $n(^{241}Pu)/n(^{239}Pu)$, $n(^{242}Pu)/n(^{239}Pu)$ are optional, but it is highly recommended also to report the minor ratios. Please be aware that the reference date for your measurements results is **1 March 2013!** For more information, please consult the participants' guidelines for result reporting annexed to this letter.

You can report the results via the following website:

https://web.jrc.ec.europa.eu/ilcReportingWeb

To access this website you need your personal password key, which is:

«Part_key»

Retieseweg 111, 2440 Geel, Belgium
Tel.: +32-(0)14-571 623 • Fax: +32-(0)14-571 863
jrc-irmm-nusimep@ec.europa.eu • http://irmm.jrc.ec.europa.eu/

The system will guide you through the reporting procedure. The result-reporting page will be active from 1st August 2012. After entering your results, please also complete the questionnaire. Do not forget to submit and always confirm when required. Directly after submitting your results and filling out the questionnaire online, you will be prompted to print the completed report form. Please do so, sign the paper version and return it to IRMM by fax (+32 14 571 863) or by e-mail. Check your results carefully for any errors before submission, since this is your definitive confirmation.

The deadline for submission of results is 1 April 2013.

Please do not hesitate to contact us in case you need more information.

Yours sincerely,

Renáta Buják
NUSIMEP-8 Co-ordinator

Yetunde Aregbe
IRMM Safeguards Co-ordinator

Annexes:

- Participants' guidelines for reporting
- Confirmation of sample receipt
- Checklist

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Annex 5: Questionnaire

Milc questionnaire

Comparison for NUSIMEP-8

Please complete this form together with the result reporting form. All answers will be treated confidentially (non-disclosure of the identity of the laboratories).

Submission Form

1. What is the mission of your laboratory (you can make more than one choice)?

- ☐ a) Environmental sciences
- ☐ b) Network of analytical laboratories (NWAL)
- ☐ c) Measurements for fissile material control or safeguards
- ☐ d) Other

1.1. If other, please specify:

2. Is your laboratory certified, accredited or authorised for this type of analysis (you can make more than one choice)?

- ☐ a) Certified
- ☐ b) Accredited
- ☐ c) Authorised

3. Is your laboratory working according to a quality management system?

- ○ no
- ○ yes

3.1. If yes, please specify: *

- ☐ ISO 17025
- ☐ ISO 9000 series
- ☐ Other

3.1.1. If other, please specify:

4. Does your laboratory participate in inter-laboratory comparisons?

- ○ no
- ○ yes

- Page 1 of 6 -

4.1. If yes, please list the name(s) and the organizer(s): *

5. How many measurements of this type does your laboratory routinely perform per year?

- ◯ 0-10
- ◯ 11-50
- ◯ 51-100
- ◯ > 100

6. How does your laboratory rate itself for these types of measurement?

- ◯ Experienced
- ◯ Less experienced
- ◯ Not experienced

7. In what type of matrices do you routenily measure uranium and/or plutonium isotope ratios (you can make more than one choice)?

- ☐ a) Sediments
- ☐ b) Soils
- ☐ c) Urine
- ☐ d) Sea water
- ☐ e) Surface water
- ☐ f) Drinking water
- ☐ g) Biota
- ☐ h) Other

7.1. If Other, please specify:

8. Was the NUSIMEP-8 sample treated according to the same analytical procedure routinely used in your laboratory?

- ◯ no
- ◯ yes

8.1. If no, please specify why not: *

9. **Did you perform a chemical separation prior to isotope ratio measurements?**

- ◯ no
- ◯ yes

9.1. If yes, please specify and give details of the resin and reagents used: *

10. **Did you report the values for isotope ratios for 1 March 2013 when submitting the results?**

- ◯ no
- ◯ yes

11. **If you used alpha-spectrometry to measure isotope ratios, which source preparation technique did you apply?**

- ◯ applicable
- ◯ not applicable

11.1. If applicable, please choose *

- ◯ a) Electrodeposition
- ◯ b) Rare earth co-precipitation
- ◯ c) Drop deposition
- ◯ d) Other

11.1.1. If other, please specify *

12. **If you used alpha-spectrometry to measure isotope ratios, please describe the equipment and detector:**

- ◯ applicable
- ◯ not applicable

12.1. Equipment *

12.2. Detector *

13. **If you used a mass-spectrometric technique to measure isotope ratios, did you apply a correction for mass fractionation/mass bias?**

- ◯ applicable
- ◯ not applicable

13.1. If applicable, please choose: *

- ◯ no
- ◯ yes

13.1.1. If yes, how was the mass fractionation/mass bias determined? *

14. If you used mass-spectrometric technique to measure isotope ratios, please describe the mass-spectrometer, detector and any particular experimental parameters for the measurements:

- ◯ Not applicable
- ◯ applicable

14.1. Mass-spectrometer (type) *

14.2. Detector (SEM, Ion counters, etc.) *

14.3. Any particular experimental parameters *

15. **Did you use a technique other than alpha- or mass-spectrometry to measure isotope ratios?**

- ◯ no
- ◯ yes

15.1. If yes, please specify: *

16. **Does your laboratory routinely use certified reference materials (CRMs)?**

- ◯ no
- ◯ yes

16.1. If yes, which CRM(s) and supplier(s) *

16.2. How are the CRM(s) applied (you can make more than one choice?

- ☐ a) Validation of procedure
- ☐ b) Calibration of instrument
- ☐ c) Other

16.2.1. If other, please specify:

[ ]

17. Are you familiar with the Guides for Quantifying Measurement Uncertainty issued by the International Organisation for Standardisation (ISO, 1995) and/or EURACHEM/CITAC (2000)?

- ○ no
- ○ yes

18. Were the reported uncertainties calculated according to the above-mentioned guides?

- ○ no
- ○ yes

18.1. If yes, what did you report as an uncertainty? *

- ○ a) Standard uncertainty
- ○ b) Expanded uncertainties with a coverage factor, k: __________

18.2. If no, how were the measurement uncertainties evaluated? *

[ ]

19. Do you routinely report uncertainties on measurements to your customers?

- ○ no
- ○ yes

20. How did you learn about NUSIMEP-8 (you can make more than one choice)?

- ☐ a) IRMM website
- ☐ b) e-mail
- ☐ c) From other participants
- ☐ d) Other

20.1. If other, please specify:

[ ]

21. Would you be interested in participating in future NUSIMEP inter-laboratory comparisons?

- ○ no
- ○ yes

21.1. What type of samples would you be interested in? *

[ ]

21.2. Which isotope(s)? *

21.3. What type of matrices? *

21.4. What concentration level? *

21.5. Other, please specify: *

22. **Do you have any feedback/comments on NUSIMEP-8?**

23. **Questionnaire completed by:**

23.1. Name

23.2. Position:

Annex 6: Confirmation of sample receipt

European Commission

NUSIMEP

Geel, 13 June 2012
JRC.D.2/RB/mt/ARES(2012) 12-069/ 720934

«TITLE» «FIRSTNAME» «SURNAME»
«ORGANISATION»
«DEPARTMENT»
«ADDRESS»
«ADDRESS1»
«ADDRESS2»
«ADDRESS3»
«ZIP» «TOWN»
«COUNTRY»

NUSIMEP-8

Confirmation of receipt of one ampoule of nitrate solution sample

Please return this form at your earliest convenience.

This confirms that the sample package has arrived.

In case the package is damaged,

please state this on the form and contact us immediately.

SAMPLE CODE ....-................

ANY REMARKS ....

....

Date of package arrival ....

Signature ....

<u>**Please return this form to:**</u>

Renata Bujak
NUSIMEP-8 Co-ordinator
EC-JRC-IRMM
Retieseweg 111
B-2440 GEEL
BELGIUM
Fax : +32 14 571 863
e-mail : jrc-irmm-nusimep@ec.europa.eu

IRMM - Retieseweg 111, B-2440 Geel - Belgium. Telephone: +32 (0)14 571 211. http://irmm.jrc.ec.europa.eu
Telephone: direct line +32 (0)14 571 623. Fax: +32 (0)14 571 863. E-mail: JRC-IRMM-NUSIMEP@ec.europa.eu

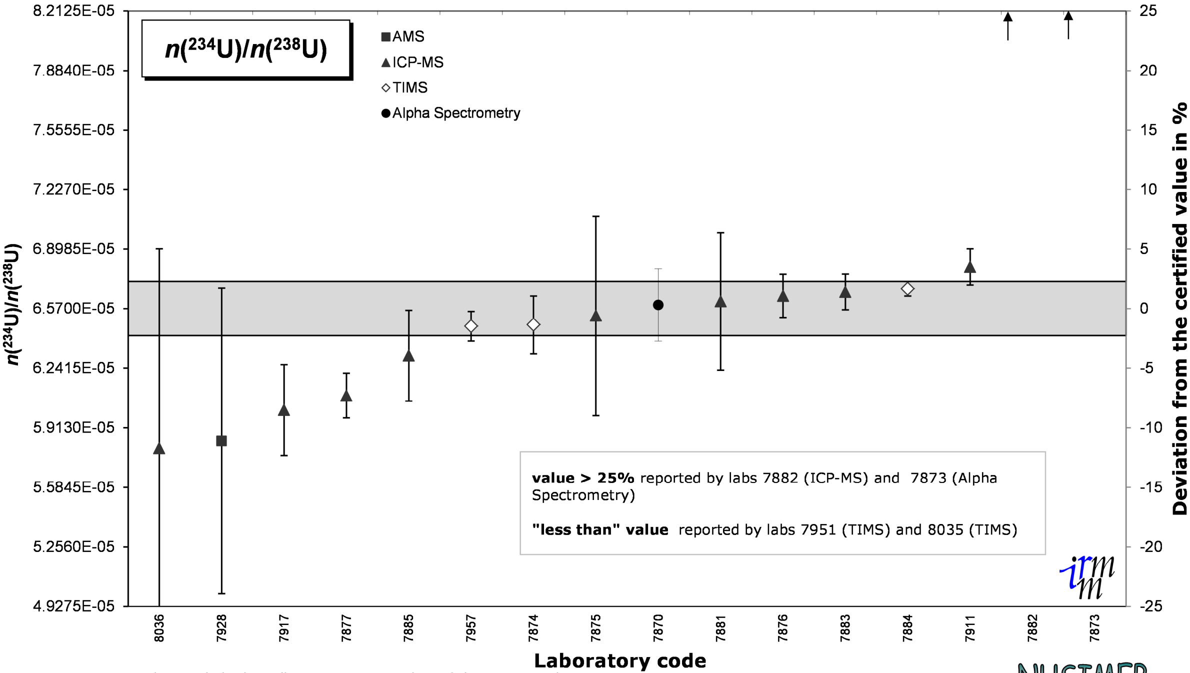
Annex 7: Results for $n(^{234}U)/n(^{238}U)$ in NUSIMEP-8

Laboratory	Analytical method	Reported $n(^{234}U)/n(^{238}U)$	Reported uncertainty $n(^{234}U)/n(^{238}U)$	Coverage factor *k*
7870	alpha spectrometry	0.0000659	0.0000165	2
7873	alpha spectrometry	1.1	0.09	2
7874	TIMS	0.0000648	0.0000016	2
7875	ICP-MS	0.0000653	0.0000055	4.3
7876	ICP-MS	0.0000664	0.0000012	2
7877	ICP-MS	0.0000609	0.00000123	2
7881	ICP-MS	0.0000661	0.0000038	2
7882	ICP-MS	0.000086	0.000001	2
7883	ICP-MS	0.00006662	0.00000099	1
7884	TIMS	0.0000668	0.0000004	2
7885	ICP-MS	0.0000631	0.0000025	2
7911	ICP-MS	0.000068	0.000001	2
7917	ICP-MS	0.0000601	0.0000025	2
7928	AMS	0.0000584	0.000008429	2
7951	TIMS	<0.002145	-	-
7957	TIMS	0.00006472	0.00000082	2
8035	TIMS	<0.001534	-	-
8036	ICP-MS	0.000058	0.000011	1

Laboratory	IAEA-SGAS-QQ: 10% z score	zeta score	IAEA-SGAS-QQ: 10% acceptable uncertainty for $\|zeta\| \leq 2$
7870	0.06	0.02	NO
7873	334835.40	24.44	-
7874	-0.27	-0.82	YES
7875	-0.12	-0.27	YES
7876	0.21	0.73	YES
7877	-1.46	-4.95	-
7881	0.12	0.20	YES
7882	6.18	22.52	-
7883	0.28	0.74	YES
7884	0.33	1.42	YES
7885	-0.79	-1.78	YES
7911	0.70	2.55	-
7917	-1.70	-3.84	-
7928	-2.22	-1.71	YES
7951	-	-	-
7957	-0.30	-1.15	YES
8035	-	-	-
8036	-2.34	-0.70	NO

NUSIMEP-8: Low-level uranium and plutonium in synthetic nitrate solution

Certified value for $n(^{234}U)/n(^{238}U)$: 0.000 065 7 ± 0.000 001 5 [$U=k \cdot u_c(k=2)$]



This graph displays all measurement results and their associated uncertainties.
These uncertainties are shown as reported, with various coverage factors and levels of confidence.
The grey band represents the reference interval ($X_{ref} \pm 2u_{ref}$).

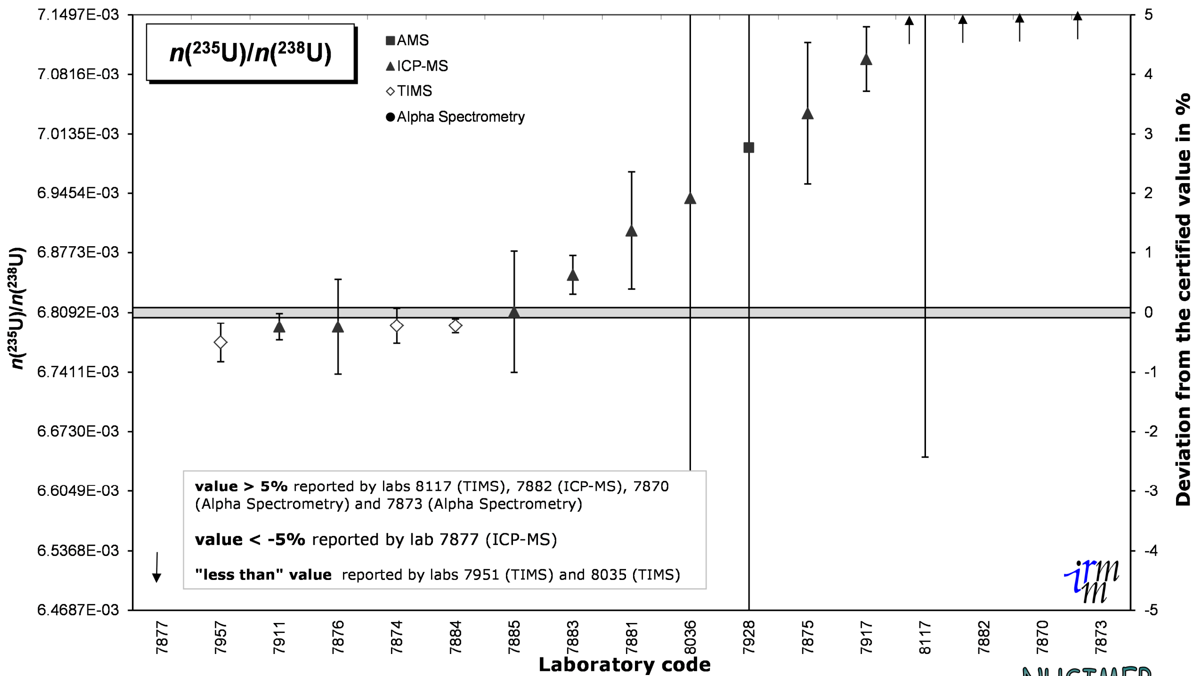
Annex 8: Results for $n(^{235}U)/n(^{238}U)$ in NUSIMEP-8

Laboratory	Analytical method	Reported $n(^{235}U)/n(^{238}U)$	Reported uncertainty $n(^{235}U)/n(^{238}U)$	Coverage factor k
7870	alpha spectrometry	0.00819	0.00287	2
7873	alpha spectrometry	0.05	0.01	2
7874	TIMS	0.006794	0.00002	2
7875	ICP-MS	0.007037	0.000081	4.3
7876	ICP-MS	0.006793	0.000054	2
7877	ICP-MS	0.00627	0.000125	2
7881	ICP-MS	0.006903	0.000067	2
7882	ICP-MS	0.007765	0.000210	2
7883	ICP-MS	0.006852	0.000022	1
7884	TIMS	0.006794	0.000008	2
7885	ICP-MS	0.00681	0.000069	2
7911	ICP-MS	0.006793	0.000015	2
7917	ICP-MS	0.007099	0.000037	2
7928	AMS	0.006998	0.000775	2
7951	TIMS	<0.00629	=	-
7957	TIMS	0.006775	0.000022	2
8035	TIMS	<0.00731	=	-
8036	ICP-MS	0.00694	0.00043	1
8117	TIMS	0.0077031	0.0010592	2

Laboratory	IAEA-SGAS-QQ: 1% z score	zeta score	IAEA-SGAS-QQ: 1% acceptable uncertainty for $\|zeta\| \le 2$
7870	40.56	0.96	NO
7873	1268.60	8.64	-
7874	-0.45	-1.46	YES
7875	6.69	11.96	-
7876	-0.48	-0.60	YES
7877	-15.84	-8.62	-
7881	2.76	2.79	-
7882	28.07	9.10	-
7883	1.26	1.93	YES
7884	-0.45	-3.09	-
7885	0.02	0.02	YES
7911	-0.48	-2.02	-
7917	8.51	15.48	-
7928	5.55	0.49	NO
7951	-	-	-
7957	-1.00	-3.01	-
8035	-	-	-
8036	3.84	0.30	NO
8117	26.26	1.69	NO

NUSIMEP-8: Low-level uranium and plutonium in synthetic nitrate solution

Certified value for $n(^{235}U)/n(^{238}U)$: 0.006 809 2 ± 0.000 005 7 [$U=k \cdot u_c(k=2)$]



This graph displays all measurement results and their associated uncertainties.
These uncertainties are shown as reported, with various coverage factors and levels of confidence.
The grey band represents the reference interval ($X_{ref} \pm 2u_{ref}$).

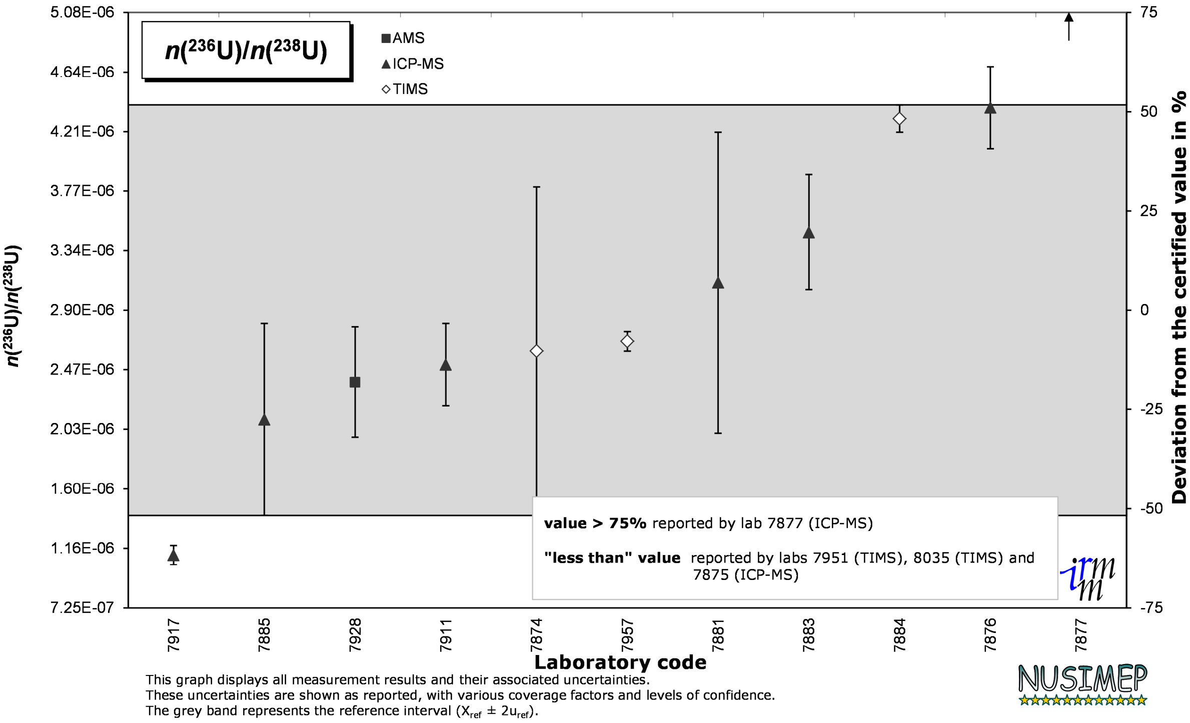
Annex 9: Results for $n(^{236}U)/n(^{238}U)$ in NUSIMEP-8

Laboratory	Analytical method	Reported $n(^{236}U)/n(^{238}U)$	Reported uncertainty $n(^{236}U)/n(^{238}U)$	Coverage factor k
7874	TIMS	0.0000026	0.0000012	2
7875	ICP-MS	<0.000017	-	-
7876	ICP-MS	0.00000438	0.0000003	2
7877	ICP-MS	0.0000162	0.00000032	2
7881	ICP-MS	0.0000031	0.0000011	2
7883	ICP-MS	0.00000347	0.00000042	1
7884	TIMS	0.0000043	0.0000001	2
7885	ICP-MS	0.0000021	0.0000007	2
7911	ICP-MS	0.0000025	0.0000003	2
7917	ICP-MS	0.00000111	0.00000007	2
7928	AMS	0.000002373	0.000000402	2
7951	TIMS	<0.000173	-	-
7957	TIMS	0.00000267	0.00000007	2
8035	TIMS	<0.00018	-	-

Laboratory	IAEA-SGAS-QQ: 10% z score	zeta score	IAEA-SGAS-QQ: 10% acceptable uncertainty for $\lVert zeta \rVert \leq 2$
7874	-2.07	-0.31	NO
7875	-	-	-
7876	10.21	1.94	YES
7877	91.72	17.34	-
7881	1.38	0.22	NO
7883	3.93	0.66	NO
7884	9.66	1.86	YES
7885	-5.52	-0.97	NO
7911	-2.76	-0.52	YES
7917	-12.34	-2.38	-
7928	-3.63	-0.68	YES
7951	-	-	-
7957	-1.59	-0.31	YES
8035	-	-	-

NUSIMEP-8: Low-level uranium and plutonium in synthetic nitrate solution

Certified value for $n(^{234}U)/n(^{238}U)$: 0.000 002 9 ± 0.000 001 5 [$U=k \cdot u_c(k=2)$]

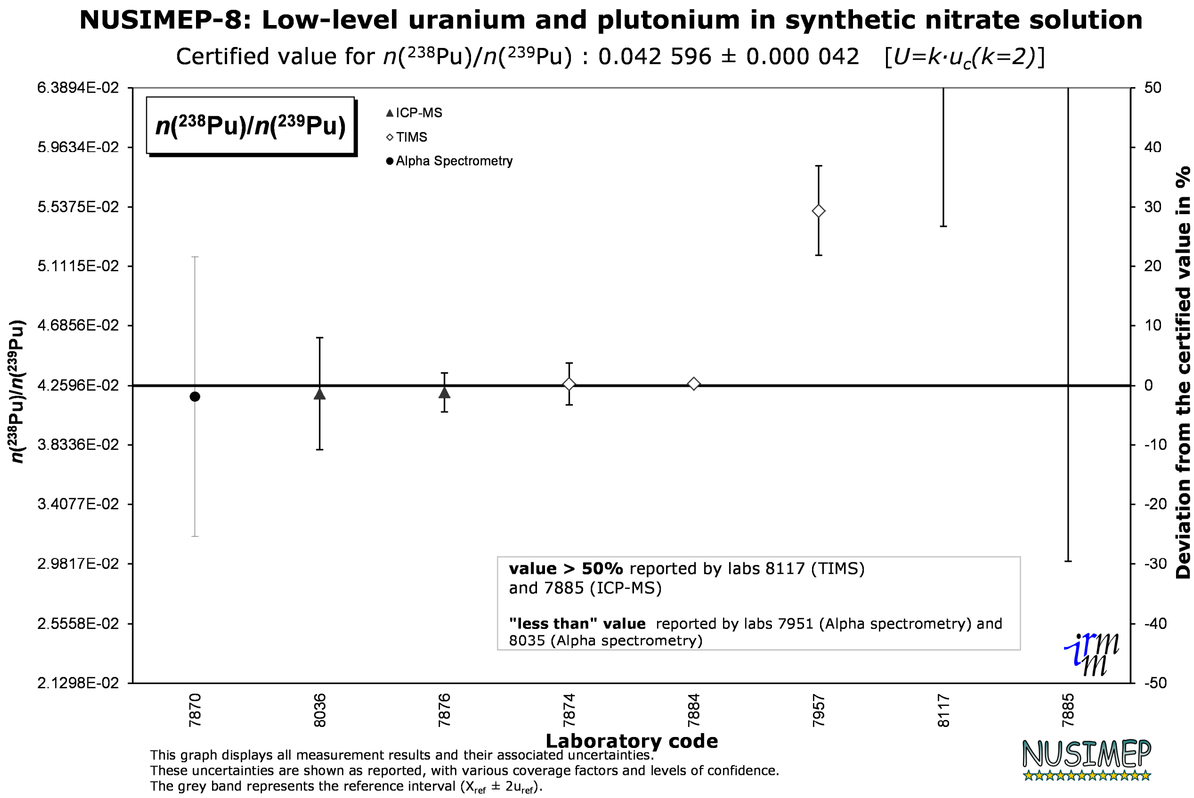


This graph displays all measurement results and their associated uncertainties.
These uncertainties are shown as reported, with various coverage factors and levels of confidence.
The grey band represents the reference interval ($X_{ref} \pm 2u_{ref}$).

Annex 10: Results for $n(^{238}Pu)/n(^{239}Pu)$ in NUSIMEP-8

Laboratory	Analytical method	Reported $n(^{238}Pu)/n(^{239}Pu)$	Reported uncertainty $n(^{238}Pu)/n(^{239}Pu)$	Coverage factor *k*	zeta score
7870	alpha spectrometry	0.0418	0.0045	2	-0.35
7874	TIMS	0.0427	0.0015	2	0.14
7876	alpha spectrometry	0.0421	0.0014	2	-0.71
7884	TIMS	0.04273	0.00004	2	4.62
7885	ICP-MS	0.15	0.12	2	1.79
7951	alpha spectrometry	<0.064276	-	-	-
7957	TIMS	0.0551	0.0032	2	7.81
8035	alpha spectrometry	<0.060722	-	-	-
8036	alpha spectrometry	0.042	0.004	1	-0.15
8117	TIMS	0.081	0.027	2	2.84

As there are no IAEA-SGAS-QG defined for the minor uranium isotope ratios, there were no z scores and acceptable uncertainty scores issued for $n(^{238}Pu)/n(^{239}Pu)$



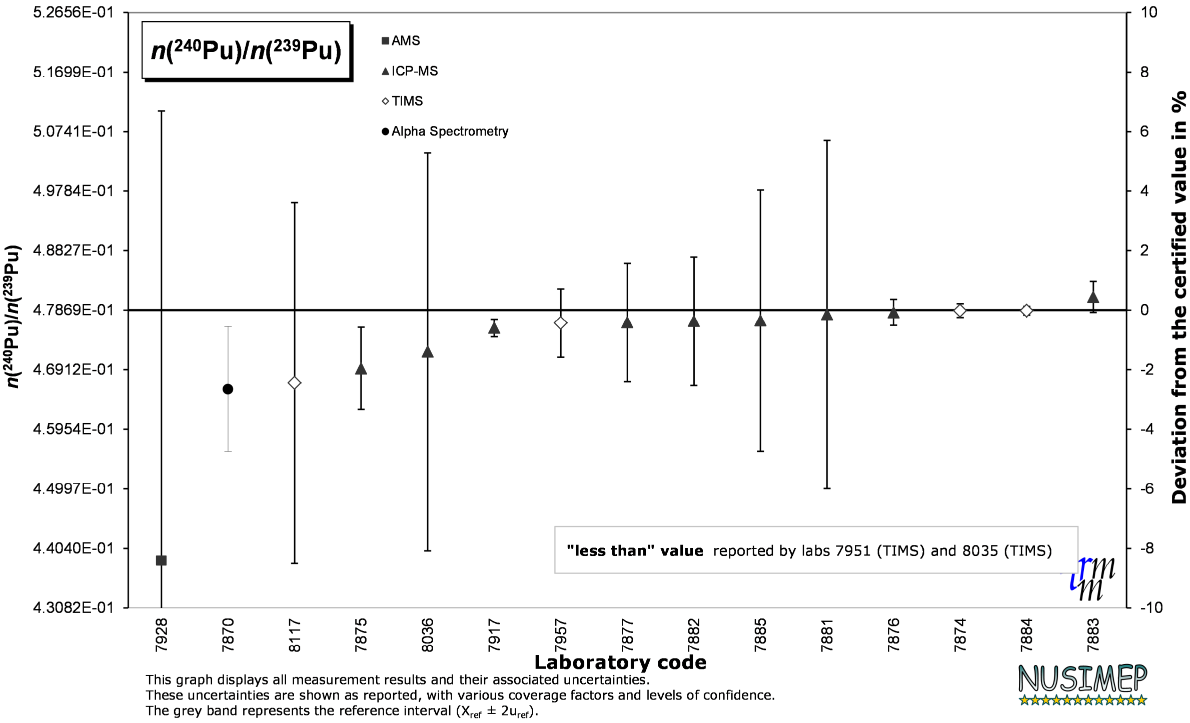
Annex 11: Results for $n(^{240}Pu)/n(^{239}Pu)$ in NUSIMEP-8

Laboratory	Analytical method	Reported $n(^{240}Pu)/n(^{239}Pu)$	Reported uncertainty $n(^{240}Pu)/n(^{239}Pu)$	Coverage factor *k*
7870	ICP-MS	0.466	0.049	2
7874	TIMS	0.4786	0.0011	2
7875	ICP-MS	0.4693	0.0066	4.3
7876	ICP-MS	0.4783	0.0021	2
7877	ICP-MS	0.4767	0.0095	2
7881	ICP-MS	0.478	0.028	2.3
7882	ICP-MS	0.4769	0.0103	2
7883	ICP-MS	0.4808	0.0025	1
7884	TIMS	0.4786	0.0007	2
7885	ICP-MS	0.477	0.021	2
7917	ICP-MS	0.4758	0.0014	2
7928	ASM	0.43839	0.07234	2
7951	TIMS	<0.440016	-	-
7957	TIMS	0.4766	0.0055	2
8035	TIMS	<0.445584	-	-
8036	ICP-MS	0.472	0.032	1
8117	TIMS	0.470	0.029	2

Laboratory	ITV: 10% z score	zeta score	ITV: 10% acceptable uncertainty for $\lVert zeta \rVert \leq 2$
7870	-0.53	-0.52	YES
7874	0.00	-0.17	YES
7875	-0.39	-6.12	-
7876	-0.02	-0.37	YES
7877	-0.08	-0.42	YES
7881	-0.03	-0.06	YES
7882	-0.07	-0.35	YES
7883	0.09	0.84	YES
7884	0.00	-0.26	YES
7885	-0.07	-0.16	YES
7917	-0.12	-4.13	-
7928	-1.68	-1.11	YES
7951	-	-	-
7957	-0.09	-0.76	YES
8035	-	-	-
8036	-0.28	-0.21	YES
8117	-0.36	-0.60	YES

NUSIMEP-8: Low-level uranium and plutonium in synthetic nitrate solution

Certified value for $n(^{240}Pu)/n(^{239}Pu)$: 0.478 692 ± 0.000 055 [$U=k \cdot u_c(k=2)$]



This graph displays all measurement results and their associated uncertainties.
These uncertainties are shown as reported, with various coverage factors and levels of confidence.
The grey band represents the reference interval ($X_{ref} \pm 2u_{ref}$).

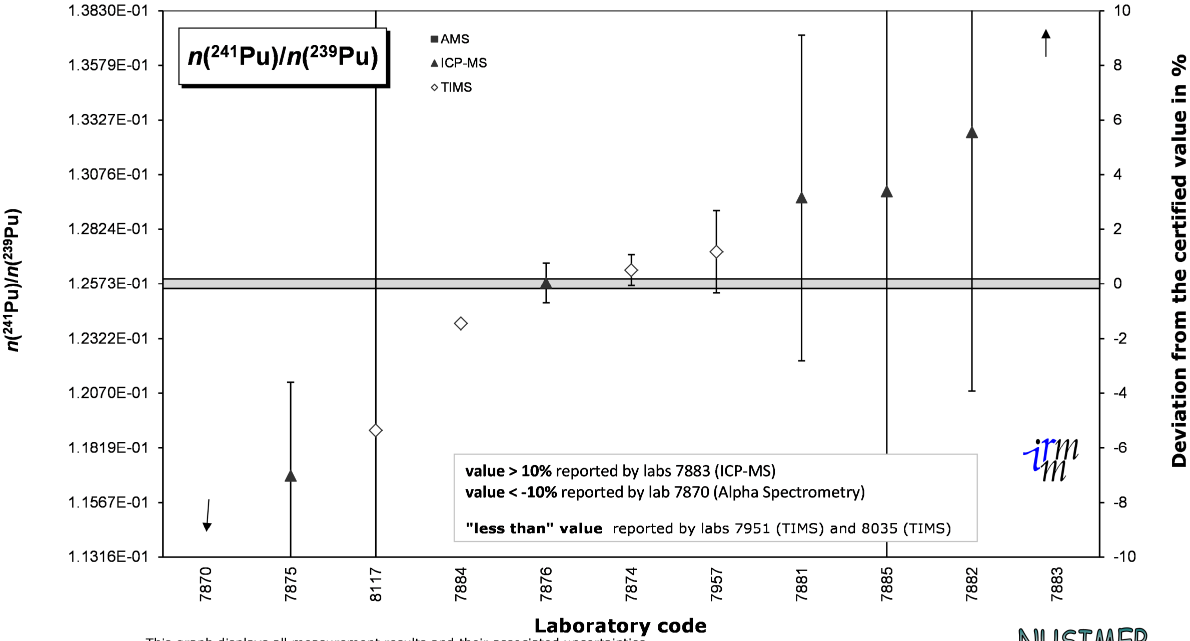
Annex 12: Results for *n*(^{241}Pu)/*n*(^{239}Pu) in NUSIMEP-8

Laboratory	Analytical method	Reported *n*(^{241}Pu)/*n*(^{239}Pu)	Reported uncertainty *n*(^{241}Pu)/*n*(^{239}Pu)	Coverage factor *k*
7870	-	0.095	0.01	2
7874	TIMS	0.12636	0.00072	2
7875	ICP-MS	0.1169	0.0043	4.3
7876	ICP-MS	0.12577	0.00092	2
7881	ICP-MS	0.1297	0.0075	2.3
7882	ICP-MS	0.1327	0.0119	2
7883	ICP-MS	0.1835	0.0011	1
7884	TIMS	0.1239	0.00004	2
7885	ICP-MS	0.13	0.09	2
7951	TIMS	<0.093582	-	-
7957	TIMS	0.1272	0.0019	2
8035	TIMS	<0.116045	-	-
8117	TIMS	0.119	0.032	2

Laboratory	ITV: 10% z score	zeta score	ITV: 10% acceptable uncertainty for $\lVert zeta \rVert \leq 2$
7870	-4.89	-6.14	-
7874	0.10	1.67	YES
7875	-1.40	-8.77	-
7876	0.01	0.08	YES
7881	0.63	1.22	YES
7882	1.11	1.17	YES
7883	9.19	52.23	-
7884	-0.29	-15.68	-
7885	0.68	0.09	NO
7951	-	--	-
7957	0.23	1.54	YES
8035	-	-	-
8117	-1.07	-0.42	NO

NUSIMEP-8: Low-level uranium and plutonium in synthetic nitrate solution

Certified value for $n(^{241}Pu)/n(^{239}Pu)$: 0.125 73 ± 0.000 23 [$U=k \cdot u_c(k=2)$]



This graph displays all measurement results and their associated uncertainties.
These uncertainties are shown as reported, with various coverage factors and levels of confidence.
The grey band represents the reference interval ($X_{ref} \pm 2u_{ref}$).

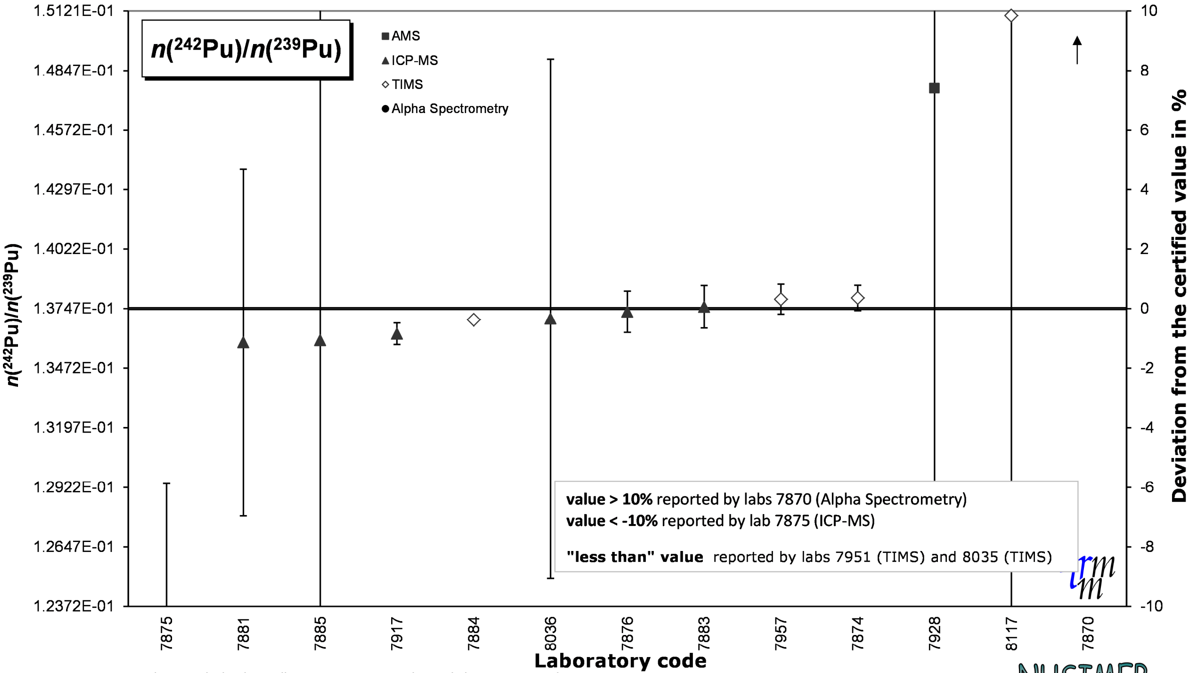
Annex 13: Results for $n(^{242}Pu)/n(^{239}Pu)$ in NUSIMEP-8

Laboratory	Analytical method	Reported $n(^{242}Pu)/n(^{239}Pu)$	Reported uncertainty $n(^{242}Pu)/n(^{239}Pu)$	Coverage factor *k*
7870	alpha spectrometry	0.536	0.072	2
7874	TIMS	0.13796	0.00059	2
7875	ICP-MS	0.1227	0.0067	4.3
7876	ICP-MS	0.13732	0.00096	2
7881	ICP-MS	0.1359	0.008	2.3
7883	ICP-MS	0.13755	0.00099	1
7884	TIMS	0.13694	0.00007	2
7885	ICP-MS	0.136	0.016	2
7917	ICP-MS	0.1363	0.0005	2
7928	AMS	0.14765	0.02016	2
7951	TIMS	<0.103474	-	-
7957	TIMS	0.1379	0.0007	2
8035	TIMS	<0.119712	-	-
8036	ICP-MS	0.137	0.012	1
8117	TIMS	0.151	0.028	2

Laboratory	ITV: 10% z score	zeta score	ITV: 10% acceptable uncertainty for $\|zeta\| \leq 2$
7870	57.98	11.07	-
7874	0.07	1.66	YES
7875	-2.15	-9.48	-
7876	-0.02	-0.31	YES
7881	-0.23	-0.45	YES
7883	0.01	0.08	YES
7884	-0.08	-13.26	-
7885	-0.21	-0.18	YES
7917	-0.17	-4.66	-
7928	1.48	1.01	YES
7951	-	-	-
7957	0.06	1.23	YES
8035	-	-	-
8036	-0.07	-0.04	YES
8117	1.97	0.97	YES

NUSIMEP-8: Low-level uranium and plutonium in synthetic nitrate solution

Certified value for $n(^{242}Pu)/n(^{239}Pu)$: 0.137 468 ± 0.000 038 [$U=k \cdot u_c(k=2)$]



This graph displays all measurement results and their associated uncertainties.
These uncertainties are shown as reported, with various coverage factors and levels of confidence.
The grey band represents the reference interval (X_{ref} ± $2u_{ref}$).

Annex 14: Summary of the information given by the participants on instrument parameters and measurement approaches

Laboratory	What is the mission of your laboratory?
7870	Environmental sciences
7873	Environmental sciences, measurements for fissile material control or safeguards
7874	NWAL, measurements for fissile material control or safeguards
7875	NWAL, Environmental sciences
7876	Research and development
7877	NWAL
7881	NWAL
7882	Analysis of nuclear materials for safeguards
7883	Research and development
7884	NWAL, measurements for fissile material control or safeguards
7885	NWAL
7911	Environmental sciences
7917	Environmental sciences, measurements for fissile material control or safeguards
7928	NWAL, Environmental sciences
7951	measurements for fissile material control or safeguards
7957	NWAL, measurements for fissile material control or safeguards
8035	measurements for fissile material control or safeguards
8036	NWAL, measurements for fissile material control or safeguards, environmental sciences
8117	NWAL, measurements for fissile material control or safeguards

Laboratory	Did you perform a chemical separation prior to measurement?	Which resin?
7870	YES	UTEVA, TEVA
7873	YES	UTEVA, TRU
7874	YES	UTEVA, anion exchnage
7875	YES	UTEVA, TEVA
7876	YES	UTEVA, TEVA, TRU
7877	NO	
7881	YES	AG1X4, AG1X8
7882	YES	TRU

7883	NO	
7884	YES	UTEVA
7885	YES	UTEVA
7911	NO	
7917	YES	UTEVA, TEVA
7928	YES	TEVA, UTEVA
7951	YES	UTEVA
7957	NO	
8035	YES	UTEVA
8036	YES*	UTEVA
8117	YES*	UTEVA

* for alpha measurement only, for ICP-MS no separation

Laboratory	Did you use alpha spectrometry to measure isotope ratios?	Which source preparation technique did you apply?
7870	YES	Electrodeposition
7873	YES	Rare earth coprecipitation
7874	NO	
7875	NO	
7876	YES	Electrodeposition
7877	NO	
7881	NO	
7882	NO	
7883	NO	
7884	NO	
7885	NO	
7911	NO	
7917	NO	
7928	NO	
7951	YES	Drop deposition
7957	NO	
8035	YES	Drop deposition
8036	YES	Rare earth coprecipitation
8117	YES	Drop deposition

Laboratory	Did you use a mass-spectrometric technique to measure isotope ratios?	Did you apply a correction for mass fractionation?
7870	YES*	
7873	NO	
7874	YES	Standards, linear law
7875	YES	standards
7876	YES	Standards, exponential law
7877	YES	Exponential law
7881	YES	Standards, exponential law
7882	YES	Standards, linear correction
7883	YES	Standards, Russel law
7884	YES	standards
7885	YES	Standard, bracketing
7911	YES	Standards, linear law
7917	YES	standards
7928	YES	Normalization with standards
7951	YES	NO
7957	YES	standards
8035	YES	NO
8036	YES	No infomartion
8117	YES	standards

*partially

Laboratory	Describe the mass spectrometer used?	Detector
7870	ICP-MS, Quadrupol Agilent	SEM
7873	NO	
7874	Triton TIMS, Thermo Fisher Scientific	SEM with RPQ (energy filter)
7875	QMS, Perkin-Elmer	No infomartion
7876	ICP-MS, Element XR, Thermo Finnigan	SEM
7877	MC ICP-MS	SEM
7881	ICP-MS, X-series, Element XR	SEM
7882	ICP-MS, Element 2	SEM
7883	MC-ICP-MS, Neptune, Thermo Fisher	SEM
7884	Triton+ TIMS	SEM
7885	ICP-MS, Element XR	Ion counters
7911	ICP-MS, Thermo Element 2	SEM
7917	MC-ICP-MS, Isoprobe	Faraday, daly SEM
7928	AMS	Gas ionisation detector

7951	TIMS, ICP-MS, Sector 54-30, VG 54-10	Faraday, Daly
7957	TIMS, Isoprobe	SEM
8035	TIMS, ICP-MS, Sector 54-30, VG 54-10	Faraday, Daly
8036	ICP-MS, Quadrupole	SEM
8117	TIMS, VG Isomass 54E	Faraday cup

Laboratory	**How did you estimate measurement uncertainty?**
7870	GUM
7873	GUM
7874	GUM
7875	GUM
7876	GUM
7877	GUM
7881	GUM
7882	GUM
7883	GUM
7884	GUM
7885	GUM
7911	Standard deviation based on 3 replicates, k = 2
7917	GUM
7928	GUM
7951	Standard deviation, precision on duplicate pair, method QC
7957	GUM
8035	Method validation, precision on duplicate pair, method QC
8036	GUM
8117	GUM

Laboratory	**Does your laboratory routinely use CRMs?**	**CRMs and suppliers**
7870	YES	U-232, Pu-242, Am-243 NIST
7873	NO	
7874	YES	IRMM and NBL CRMs
7875	YES	U050, U0002, U020A, CRM-111A, CRM128, CRM137, CRM126, CRM130
7876	YES	IRMM, NIST, IAEA
7877	YES	CRM U500, CRMU20-A, CRM-111A
7881	YES	IRMM184, IRMM183, NBS005
7882	YES	IRMM-184, IRMM-187, CRM U030A NBL

7883	YES	NBL for U, IRMM for U and Pu
7884	YES	U030, U010, U005, IRMM085A
7885	YES	NIST, IRMM
7911	YES	IRMM 3183, 3184, 3185, NBL U005, U010, U015, 111A
7917	YES	IRMM-290, CRM 005A, 030, 200 and 350 NBL
7928	YES	IRMM 290B, 290C, NBL U005, U010, NIST 4330B 4333A
7951	YES	CRM 137 NBS 020, NBL
7957	YES	U010, U005a, CRM126a
8035	YES	CRM 137, NBS 020, NBL
8036	YES	IAEA 327
8117	YES	NBS 500, 005, 020, 050, 350, 750, 930, 960, NBS 947, IRMM081a

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