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REIMEP-17: Plutonium and uranium amount content, and isotope amount ratios in synthetic input solution

Inter-laboratory comparison, Report to participants

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Abstract
Nuclear safeguards is based on international agreements and in the EU has the rank of European law (Euratom Treaty, Chapter VII, Euratom regulation 302/2005) ensuring that dual use materials– such as uranium and plutonium – are used for peaceful purposes only. Physical verification measurements at nuclear facilities, such as reprocessing and fuel fabrication plants, are a part of safeguards inspections. The reliability of measurement results in nuclear material accountancy and verification is indispensable for an effective safeguards system. A new external quality control campaign, REIMEP-17 on "Plutonium and uranium amount content, and isotope amount ratios in synthetic input solution" was organised by JRC-IRMM in cooperation with JRC-ITU, particularly for EURATOM safeguards (DG ENER) laboratories and the IAEA Network of Analytical Laboratories for nuclear material analysis (IAEA-NWAL), as well as for laboratories from industry and experts in the field. Participating laboratories in REIMEP-17 received two samples with undisclosed U, Pu amount content and $n(^{238}\mathrm{Pu})/n(^{239}\mathrm{Pu})$, $n(^{240}\mathrm{Pu})/n(^{239}\mathrm{Pu})$, $n(^{241}\mathrm{Pu})/n(^{239}\mathrm{Pu})$ $n(^{242}\mathrm{Pu})/n(^{239}\mathrm{Pu})$ 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 ratio values. One of the samples, REIMEP-17A had uranium and plutonium amount contents typical for undiluted spent nuclear fuel input solution and the other sample, REIMEP-17B was a diluted fraction of it. The participants were requested to apply their standard analytical procedures and report the results with the associated uncertainties. The laboratories were also requested to complete and return a questionnaire so that an overall picture of the laboratories' capabilities could be made. REIMEP-17 was announced to participants in April 2012. Sixteen laboratories registered for REIMEP-17. Due to delays in the shipment of the samples and problems with the transport containers, three laboratories were not able to receive the samples. Three laboratories withdrew their participation. Consequently, the deadline for submitting the results had to be extended until July 1, 2013. In the end JRC-IRMM received results from nine laboratories; one laboratory did not submit the results. The reported measurement results have been evaluated against the independent reference values by means of z-scores and zeta-scores in compliance with international guidelines. In general the REIMEP-17 participants' results were satisfactory and in compliance with the International Target Values for Measurement Uncertainties in Safeguarding Nuclear Materials (ITV2010). This report presents the REIMEP-17 participants' results; including the evaluation of the questionnaire.

REIMEP-17: Plutonium and uranium amount content, and isotope amount ratios in synthetic input solution

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June 2014

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Summary

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1. Introduction

Nuclear safeguards aims at the verification of the non-diversion of fissile material from its intended and declared peaceful use in line with the Treaty on the non-Proliferation of Nuclear weapons (NPT) and the EURATOM Treaty. In order to reach this goal a reliable nuclear material accountancy system has to be established by the plant operator and at the same time a reliable verification system by the safeguards authority in charge. Safeguarding nuclear material involves the quantitative verification of fissile material by independent measurements. Nowadays, laboratories carrying out nuclear measurements have implemented rigorous quality control concepts and are required to demonstrate their measurement capabilities in compliance with The International Target Values for Measurement Uncertainties in Safeguarding Nuclear Materials (ITV2010) on a regular and timely basis to legal and safeguards authorities [1]. This also includes participation in inter-laboratory comparisons.

The JRC-IRMM is an accredited provider of inter-laboratory comparisons according to ISO/IEC 17043:2010 with a long experience in organising quality control campaigns for measurement of nuclear samples for safeguards and fissile material control [2]. The Regular European Inter-laboratory Measurement Evaluation Programme (REIMEP) was established in 1982 as an external quality control tool for measurement of uranium and plutonium amount contents and isotope ratios in various samples of the nuclear fuel cycle; controlled by nuclear safeguards authorities. Previous REIMEP inter-laboratory comparisons have included samples such as uranium oxide, uranium in nitric acid, uranium in the form of UF_6, plutonium oxide, and others [3].

REIMEP-17 is focused on plutonium and uranium amount content, and isotope amount ratios in synthetic input solutions. It was announced for participation on April 1, 2012. Participants in REIMEP-17 received two sample solutions with undisclosed U and Pu amount contents and isotope amount ratios. The samples were prepared from mixed oxide fuel in nitric solution and the addition of natural uranium. The preparation as well as the shipment of the test samples to the participants was carried out by JRC-ITU. The reference values were established by isotope dilution mass spectrometry (IDMS) and thermal ionisation mass spectrometry (TIMS) at JRC-IRMM.

The participants were requested to measure the measurands specified using their routine analytical procedures and report measurement results with associated uncertainties. The original submission deadline of April 1, 2013 had to be extended to July 1, 2013 due to problems with the shipping containers and delays in the transport of the samples.
Participating laboratories in REIMEP can compare their measurement results with independent and traceable reference values obtained by measurements applying the principles of metrology. The participant results were evaluated against the reference values by means of z-scores and zeta-scores in compliance with ISO 13528:2005 [4]. The International Target Values for Measurement Uncertainties in Safeguarding Nuclear Materials (ITVs) were used as a criterion for the evaluation of the participant results. In 2010, the IAEA together with the European Safeguards Research and Development Association (ESARDA), international standardisation organisations (ISO) and regional safeguards authorities published a revised version of the ITVs [1]. The uncertainties in ITV2010 are to be considered in judging the reliability of analytical techniques applied to industrial nuclear and fissile material that are subject to safeguards verification. They should be achievable under the conditions normally encountered in typical industrial laboratories or during actual safeguards inspections. The

ITVs are intended to be used by nuclear plant operators and safeguards organisations as a reference of the quality of measurements necessary for nuclear material accountancy.

The participants were also asked to complete a questionnaire when submitting the results. The aim of the questionnaire was to extract information with respect to the measurement protocols applied in their laboratories, evaluation of measurement uncertainties, type of the instrumentation, etc. and to identify future needs for inter-laboratory comparisons.

2. Scope and aim

Reliable measurements of nuclear materials are required in context of verification measures of a state's nuclear activities according to international and regional safeguards agreements. Measurements of amount contents and isotope ratios, in particular of uranium and plutonium in samples taken from proliferation-sensitive stages of the nuclear fuel cycle such as enrichment and reprocessing of nuclear fuel are of major importance. For that reason the JRC-IRMM and JRC-ITU joined efforts to provide to EURATOM and IAEA safeguards laboratories, nuclear plant operators and nuclear material laboratories REIMEP-17 on 'Plutonium and uranium amount content, and isotope amount ratios in synthetic input solution'.

For this inter-laboratory comparison, two sample solutions were prepared with different uranium and plutonium amount contents. REIMEP-17A was supplied in $3\ \mathrm{mol \cdot L^{-1}}$ nitric solution with a U, Pu concentration typical for undiluted input solutions. REIMEP-17B was a diluted fraction thereof and was supplied in $8\ \mathrm{mol \cdot L^{-1}}$ nitric solution. Both samples were delivered in laser sealed glass ampoules containing about 7 mL of solution. The measurands were the U and Pu amount content and $n(^{238}\mathrm{Pu})/n(^{239}\mathrm{Pu})$, $n(^{240}\mathrm{Pu})/n(^{239}\mathrm{Pu})$, $n(^{241}\mathrm{Pu})/n(^{239}\mathrm{Pu})$, $n(^{242}\mathrm{Pu})/n(^{239}\mathrm{Pu})$ 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. The laboratories were asked to apply their routine analytical procedure and report the results together with the measurement uncertainties and the completed questionnaire. The accompanying letter with the participation code, the guidelines on result reporting, the sample receipt form, and a checklist were also delivered together with the sample.

3. Time frame

REIMEP-17 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 shipped between July and December 2012 from JRC-ITU Karlsruhe. Due to difficulties with the sample containers and nuclear material transport issues it was not possible to ship the samples to three of the laboratories registered as participants in REIMEP-17. In addition, three laboratories withdrew their participation. For the above mentioned reason, the original reporting deadline of April 1, 2013 had to be extended to July 1, 2013 in order to give all the laboratories enough time to carry out the measurements and report the results. The certification and homogeneity assessment were carried out at JRC-IRMM in December 2012; the short-term stability assessment was finalised in August 2013. The REIMEP-17 reference values were sent to the participants on October 2, 2013.

4. Test material

4.1. Preparation of the solution

The mother solution for REIMEP-17A was prepared by dissolution of a mixed oxide fuel in nitric acid with addition of natural uranium, aiming at concentration of uranium and plutonium of about 200 mg g^{-1} and 2 mg·g^{-1}, respectively. The amount content of the uranium and plutonium and the isotopic composition in REIMEP-17A was verified by Isotope Dilution Mass Spectrometry (IDMS) at JRC-ITU. The REIMEP-17B solution was prepared by a 400-fold dilution of REIMEP-17A resulting in a concentration of uranium of about 500 µg·g^{-1} and plutonium of about 5 µg·g^{1}. The solutions were dispensed into glass ampoules with a peristaltic pump and sealed by a laser. 70 ampoules of REIMEP-17A and 70 ampoules of REIMEP-17B were prepared each containing about 7 mL of solution. The dispensing and sealing of an ampoule of REIMEP-17B are shown in Figure 1.

Figure 1: Dispensing of a REIMEP-17B sample with a peristaltic pump inside the glove box (left) and sealing of an ampoule (right) at JRC-ITU Karlsruhe.

4.2. REIMEP-17 value assignment

The reference values for the plutonium and uranium amount content were established by Isotope Dilution Thermal Ionisation Mass Spectrometry (ID-TIMS) and for the plutonium and uranium isotope amount ratios by TIMS at JRC-IRMM. The design of the study was such that the measurements for the reference value assignment and the homogeneity assessment were combined. Five ampoules from each fraction (REIMEP-17A and REIMEP-17B) were selected randomly at JRC-ITU, and subsequently analysed at JRC-IRMM. Blend mixtures were prepared by spiking with a mixed $^{233}U/^{242}Pu$ isotopic reference material (IRMM-046b). Uranium and plutonium were separated and purified by anion exchange (AG1X4, 100-200 mesh, BioRad). Isotope ratio measurements were performed on a Triton TIMS (Thermo Fisher Scientific, Bremen, Germany) by total evaporation technique [5, 6]. Three replicates were measured from each blend mixtures. IRMM-290/A3 plutonium isotopic standard solution and IRMM-074/10 uranium isotopic standard solution were measured together with the samples to correct for mass fractionation effects. The target relative standard uncertainty for method repeatability in REIMEP-17 was < 0.1% for the plutonium and uranium content and for the major (e.g. most abundant) isotope amount ratios. This goal was met for the plutonium and uranium content measured in REIMEP-17A and REIMEP-17B with a relative standard uncertainty for method repeatability ranging from 0.01% - 0.03%, for all the plutonium isotope amount ratios ranging from 0.004% - 0.1%, and for the major uranium isotope amount ratio, $n(^{235}U)/n(^{238}U)$, ranging from 0.02% - 0.04%. The relative standard uncertainty for method repeatability for the minor uranium isotope amount ratios, $n(^{234}U)/n(^{238}U)$ and $n(^{236}U)/n(^{238}U)$, was expected to be larger. It was about 1.5% for the $n(^{234}U)/n(^{238}U)$ and about 25% for the $n(^{236}U)/n(^{238}U)$, which was acceptable for the purpose of this inter-laboratory comparison. The reference value assignment for the uranium and plutonium amount content in REIMEP-17A and REIMEP-17B was done by ID-TIMS. In addition, the gravimetric preparation of the REIMEP-17A mother solution and the IDMS confirmation measurements performed at JRC-ITU together with the gravimetric dilution to REIMEP-17B provided an external verification of the reference values assigned by JRC-IRMM. The verification measurements of the mother solution of REIMEP-17A carried out at JRC-ITU confirmed the reference values within measurement uncertainties established at JRC-IRMM.

This external verification of the amount contents in the two fractions allowed a different approach for the value assignment for the uranium and plutonium isotope amount ratios. Due to the fact that it is very unlikely that isotope fractionation occurred during the gravimetric dilution of the higher concentrated fraction REIMEP-17A to the lower concentrated fraction REIMEP-17B the value assignment for the major and minor isotope amount ratios in both REIMEP-17A and REIMEP-17B was done by TIMS on the samples of the fraction REIMEP-17A and only verified in the frame of the homogeneity and stability assessment for REIMEP-17B, see also paragraph 4.3 and 4.4.

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 the type of the certified test samples provided in REIMEP-17, the homogeneity assessment was done in compliance with ISO Guide 35:2006 [7] and the IUPAC International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories [8]. 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 [7]. Furthermore, in Annex B of ISO 13528 it is stated that the number of units can be less than 10 if suitable data are available from previous homogeneity studies on similar samples prepared by the same procedure [4, 9]. The measurement results of the ten samples (five samples REIMEP-17A and five samples REIMEP-17B) were evaluated by a one-way analysis of variance (ANOVA) [10 ,11, 12]. This allows the separation of the method variation (s_{wb}) from the experimental averages over the replicates measured in one bottle to obtain an estimation for the real variation between bottles (s_{bb}), with u^*_{bb} being the lower limit of 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 [10, 11, 12] 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 [4, 7]. 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}) $\leq 0.3\,\hat{\sigma}$. The results of the homogeneity assessment in REIMEP-17A and REIMEP-17B are listed in Annex 1 and Annex 2.

The standard deviation for proficiency assessment was set in compliance with the ITV2010 relative combined standard uncertainties as follows [1]:

- *Uranium/Plutonium Element Concentration Measurements applying IDMS for all materials typically encountered in the nuclear fuel cycle under conditions of sufficiently different isotopic compositions of spike and sample and near optimum sample to spike ratio, using large size spikes (such as LSD) under hot cell conditions*
- *Uranium/Plutonium Element Concentration Measurements applying IDMS for all materials typically encountered in the nuclear fuel cycle under conditions of sufficiently different isotopic compositions of spike and sample and near optimum sample to spike ratio, using large size spikes (such as LSD) under glove box conditions*
- *Uranium/Plutonium Element Concentration Measurements applying IDMS for all materials typically encountered in the nuclear fuel cycle Under conditions of sufficiently different isotopic compositions of spike and sample and near optimum sample to spike ratio, using small size spikes under glove box conditions*
- *^{235}U Abundance Measurements applying TIMS U (0.3% <^{235}U <1%*)
- *Plutonium Isotope Assay of Pu and U/Pu Materials – high-burnup Pu*

The ITV2010 vary depending on the instrumental/analytical technique applied, and as can be seen from above, in the case of IDMS, also on the type of spike used and if the measurement is carried out under glove box or hot cell conditions. As the participants were asked to apply their routine measurement protocols to measure the REIMEP-17 samples, it was decided to use for the homogeneity and short term stability assessment the most stringent criteria for $\hat{\sigma}$ in compliance with the ITV2010. Furthermore, there are no ITVs for the minor uranium isotope ratios. Therefore, $\hat{\sigma}$ for $n(^{234}\mathrm{U})/n(^{238}\mathrm{U})$ and $n(^{236}\mathrm{U})/n(^{238}\mathrm{U})$ was derived as standard deviation from the participant results. The variation between units (s_{bb}) for all parameters under investigation in REIMEP-17A and REIMEP-17B are listed in Table 1 and Table 2. The tests indicate that the REIMEP-17A and REIMEP-17B test materials are sufficiently homogeneous for the uranium and plutonium amount contents and for the major uranium and plutonium isotope amount ratios. As can be seen from Table 1 and Table 2, for some of the minor ratios the homogeneity test resulted in a s_{bb} (or u^*_{bb}) > $0.3\hat{\sigma}$. In REIMEP-17A the $n(^{241}\mathrm{Pu})/n(^{239}\mathrm{Pu})$ amount ratios homogeneity test was not successful but s_{bb} was still considerable smaller than the respective ITV2010. In addition the stability test, and at a later stage the results from the participants in REIMEP-17A, confirmed the REIMEP-17A reference value for $n(^{241}\mathrm{Pu})/n(^{239}\mathrm{Pu})$ amount ratio within expanded uncertainty. For REIMEP-17B the uranium amount content and the minor plutonium and uranium isotope amount ratios resulted in s_{bb} (or u^*_{bb}) > $0.3\hat{\sigma}$ but again all the values for s_{bb} were considerably smaller than the respective ITV2010, except for $n(^{241}\mathrm{Pu})/n(^{239}\mathrm{Pu})$ where the relative s_{bb} exceeded the respective ITV2010. However, even for this ratio as well as for all the other minor ratios the stability measurements and the participants results confirmed the REIMEP-17B reference values within expanded uncertainties. As a result the REIMEP-17A and REIMEP-17B test materials were considered sufficiently homogeneous for the purpose of this inter-laboratory comparison and the stringent assessment criteria were not changed.

4.4. Stability

Due to the fact that transport of nuclear material can take weeks or even months, depending on licenses and shipment requirements for different countries, the 'short term' stability assessment was carried out one year after the preparation of the REIMEP-17A and REIMEP-17B samples with the aim of confirming the reference values. The samples selected for short term stability assessment were stored at room temperature and analysed by ID-TIMS and TIMS at JRC-IRMM. Methods to assess whether an ILC material is sufficiently stable for its purpose are described in ISO 13528 [4]. 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 compared to the standard deviation for proficiency assessment $\hat{\sigma}$. 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 and Table 2, the criterion was only met for the stability of the plutonium amount content in REIMEP-17B, the other results on the amount contents slightly exceeded $0.3\hat{\sigma}$. Nevertheless, there was no significant difference between the REIMEP-17 reference values and the results from the short term stability measurements within their expanded uncertainties for the uranium and plutonium amount content in REIMEP-17A and for the uranium amount content in REIMEP-17B. The short term stability measurements for all the uranium and plutonium isotope amount ratios in REIMEP-17A met the assessment criterion for a stability check, except for the minor isotope amount ratio, $n(^{234}U)/n(^{238}U)$, that slightly exceeded $0.3\hat{\sigma}$. But also for this ratio there was no significant difference between the REIMEP-17A reference value and the result from the short term stability measurement within expanded uncertainty. A significant difference was observed for the minor $n(^{242}Pu)/n(^{239}Pu)$ ratio when measuring the REIMEP-17B stability sample but the percentage difference from the REIMEP-17 certified value is considerably smaller than the respective ITV2010. All the other plutonium ratios were tested successfully for short term stability in this sample. For all the uranium ratios the $|x_s - y_s|$ was larger than $0.3\hat{\sigma}$, but as in the previous cases there was no significant difference between the REIMEP-17B reference value and the results on the uranium isotope amount ratios from the short term stability measurements within expanded uncertainties. The results of the stability assessment in REIMEP-17A and REIMEP-17B are listed in Annex 3 and Annex 4. All the relative expanded uncertainties (coverage factor, k = 2) of the measurement results performed for the short term stability were considerably smaller than the respective ITV2010, which are expressed as relative combined standard uncertainties (coverage factor, k = 1). Although the stability test criterion from ISO 13528, which does not take any measurement uncertainty into account, could not be met for all the measurands under investigation, all the amount contents and isotope amount ratios in REIMEP-17 were confirmed with expanded uncertainties in the frame of this short term stability assessment. Therefore it was concluded that the REIMEP-17A and REIMEP-17B test materials were found to be appropriate for the purpose of this inter-laboratory comparison.

The results from the homogeneity and stability assessment are summarized in Table 1 and Table 2.

Table 1: Homogeneity and stability tests for REIMEP-17A according to ISO 13528 [4]

REIMEP-17A	*Relative* s_{bb}	standard deviation for proficiency assessment $\hat{\sigma}$	Homogeneity check $s_{bb} \leq 0.3\hat{\sigma}$	Stability check $\lvert x_s - y_s \rvert \leq 0.3\hat{\sigma}$
Plutonium amount content	0.017%	$0.0009X_{ref}$	YES	NO(**)
Uranium amount content	0.013%	$0.0009X_{ref}$	YES	NO(**)
$n(^{234}U)/n(^{238}U)$	0.694%	$0.0376X_{ref}$	YES	NO(**)
$n(^{235}U)/n(^{238}U)$	0.035%	$0.0014X_{ref}$	YES	YES
$n(^{236}U)/n(^{238}U)$	5.94%	$0.44X_{ref}$	YES	YES
$n(^{238}Pu)/n(^{239}Pu)$	0.042%	$0.009X_{ref}$	YES	YES
$n(^{240}Pu)/n(^{239}Pu)$	0.004%	$0.00055X_{ref}$	YES	YES
$n(^{241}Pu)/n(^{239}Pu)$	0.089%	$0.0014X_{ref}$	NO(*)	YES
$n(^{242}Pu)/n(^{239}Pu)$	0.0046%	$0.0018X_{ref}$	YES	YES

(*) Since the relative s_{bb} was considerably smaller than the respective ITV2010 the REIMEP-17A test material was considered sufficiently homogeneous for the purpose of this inter-laboratory comparison.

(**) Since the percentage difference between the REIMEP-17A reference value and the value from short term stability testing was < 0.08% and there was no significant difference observed within expanded uncertainties, the REIMEP-17A test material was considered sufficiently stable for the purpose of this inter-laboratory comparison.

Table 2: Homogeneity and stability tests for REIMEP-17B according to ISO 13528 [4]

REIMEP-17B	*Relative* s_{bb}	standard deviation for proficiency assessment $\hat{\sigma}$	Homogeneity check $s_{bb} \leq 0.3\hat{\sigma}$	Stability check $\lvert x_s - y_s \rvert \leq 0.3\hat{\sigma}$
Plutonium amount content	0.018%	$0.0009X_{ref}$	YES	YES
Uranium amount content	0.039%	$0.0009X_{ref}$	NO(*)	NO(**)
$n(^{234}U)/n(^{238}U)$	2.99%	$0.0645X_{ref}$	NO(*)	NO(***)
$n(^{235}U)/n(^{238}U)$	0.023%	$0.0014X_{ref}$	YES	NO(**)
$n(^{236}U)/n(^{238}U)$	11.4%	$0.25X_{ref}$	NO(*)	NO(***)
$n(^{238}Pu)/n(^{239}Pu)$	0.877%	$0.009X_{ref}$	NO(*)	YES
$n(^{240}Pu)/n(^{239}Pu)$	0.005%	$0.00055X_{ref}$	YES	YES
$n(^{241}Pu)/n(^{239}Pu)$	1.4%	$0.0014X_{ref}$	NO(*)	YES
$n(^{242}Pu)/n(^{239}Pu)$	0.094%	$0.0018X_{ref}$	NO(*)	NO(****)

(*) Since the relative s_{bb} for the minor isotope amount ratios with the exception of $n(^{241}Pu)/n(^{239}Pu)$ (see paragraph 4.3) was considerably smaller than the respective ITV2010 or the standard deviations for the ILC assessment the REIMEP-17B test material was considered sufficiently homogeneous for the purpose of this inter-laboratory comparison.

(**) Since the percentage difference between the REIMEP-17B reference values and the values from short term stability testing was 0.08% - 0.1% and there was no significant difference observed within expanded uncertainties, the REIMEP-17B test material was considered sufficiently stable for the purpose of this inter-laboratory comparison.

(***) Since the percentage difference between the REIMEP-17B reference values and the values from short term stability testing for the minor uranium isotope amount ratios was 5% and 30%, respectively and there was no significant difference observed within expanded uncertainties, the REIMEP-17B test material was considered sufficiently stable for the purpose of this inter-laboratory comparison.

(****) Since the percentage difference between the REIMEP-17B reference value and the value from short term stability testing 0.1%, the REIMEP-17B test material was considered sufficiently stable for the purpose of this inter-laboratory comparison

4.5. Distribution

The ILC test samples were dispatched to the participants as nuclear material from JRC-Karlsruhe between July 2012 and December 2012. Each participant received a package with two ampoules of REIMEP-17A and REIMEP-17B, respectively; and the accompanying papers. Laboratories were requested to provide the necessary documents in order to obtain the licence to ship the materials. Due to difficulties with the transport containers and issues related to the transport of nuclear material three of the laboratories registered as participants in REIMEP-17 could not receive the samples.

5. Participant invitation, registration and information

REIMEP-17 was announced in relevant conferences and meetings (ESARDA, INMM, CETAMA, etc.). Invitations were sent to EURATOM and IAEA safeguards laboratories, nuclear plant operators, nuclear material laboratories and others who expressed interest in participation. Participants were asked to determine the U, Pu amount content and $n(^{238}\mathrm{Pu})/n(^{239}\mathrm{Pu})$, $n(^{240}\mathrm{Pu})/n(^{239}\mathrm{Pu})$, $n(^{241}\mathrm{Pu})/n(^{239}\mathrm{Pu})$, $n(^{242}\mathrm{Pu})/n(^{239}\mathrm{Pu})$, $n(^{234}\mathrm{U})/n(^{238}\mathrm{U})$, $n(^{235}\mathrm{U})/n(^{238}\mathrm{U})$, $n(^{236}\mathrm{U})/n(^{238}\mathrm{U})$, amount ratios applying their routine procedures.

Participants were also informed that their measurement results would be evaluated against the certified reference values and that full confidentiality would be guaranteed to the link between measurement results and the participants' identity. The call for participation in REIMEP-17 was also announced on the IRMM website (Annex 5). A confirmation of registration was sent to those participants who had registered (Annex 6). The accompanying letter with the instructions was sent to the participants together with the certified test samples (Annex 7). This letter contained the individual code to access via the respective website the result reporting and related questionnaire pages (Annex 8). After sample receipt the participants returned the signed 'Confirmation of sample receipt' (Annex 9). In addition, a guide to help the participants with the online reporting tool was also provided. The number of participants per country is shown in Table 3.

Table 3: Number of participants per country

Country	Number of participants
Austria	2
Belgium	1
France	3
The Netherlands	1
Republic of Korea	1 (no shipment)
Russian federation	1 (no shipment)
Switzerland	1 (no shipment)
United Kingdom	3
United States	3 (cancelled)

6. REIMEP-17 reference values

The REIMEP-17 reference values X_{ref} and their associated expanded uncertainties U_{ref} (k=2) are shown in Table 4.

Table 4: REIMEP-17: Plutonium and uranium amount content, and isotope amount ratios in synthetic input solution reference values

REIMEP-17A	Amount content	
	Certified value [1)] [µmol/g]	Uncertainty [2)] [µmol/g]
Pu	9.1561	0.0050
U	843.42	0.50
	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
REIMEP-17B	Amount content	
	Certified value [1)] [µmol/g]	Uncertainty [2)] [µmol/g]
Pu	0.022976	0.000013
U	2.1167	0.0020
	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

Participants were asked to report the uranium and plutonium amount content and the uranium and plutonium isotope amount ratios for REIMEP-17A and REIMEP-17B. Participants were requested to report every result with an uncertainty and a coverage factor. Nine participants reported measurement results; among those seven participants submitted results for both REIMEP-17A and REIMEP-17B samples. One laboratory did not submit the results before the REIMEP-17 reference values were made public to the participants. From the submitted results, two laboratories reported only an upper limit for the $n(^{236}U)/n(^{238}U)$ amount ratio for REIMEP-17A and REIMEP-17B while one laboratory did not report a value for this minor uranium amount ratio. One laboratory reported an upper limit for the uranium and plutonium amount content in REIMEP-17B sample. All the results are displayed as they were reported by the participants.

7.2. Measurement results

The individual measurement results and display overview graphs are listed in Annexes 10-27.

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 [4].

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

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| ≤ 2, questionable result for 2 < |score| ≤ 3 and unsatisfactory result for |score| > 3.

z score

The REIMEP-17 z score indicates whether a laboratory is able to perform the measurement in accordance with the International Target Values for Measurement Uncertainties in Safeguarding Nuclear Materials. As already outlined in paragraph 4.3, the ITV2010 are different for analytical approaches and techniques applied [1]. To take this into account, two different standard deviations for proficiency assessment $\hat{\sigma}$ complying with the different ITV2010 for the measurements of uranium/plutonium amount content in the REIMEP-17 samples $\hat{\sigma}$ are listed in Table 5.

Table 5: REIMEP-17 standard deviations for proficiency assessment

REIMEP-17A / REIMEP-17B	standard deviation for proficiency assessment $\hat{\sigma}$ in compliance with ITV2010 [1]
Plutonium amount content$^{(*)}$	$0.0009X_{ref}$
Uranium amount content$^{(*)}$	$0.0009X_{ref}$
Plutonium amount content$^{(**)}$	$0.0014X_{ref}$
Uranium amount content$^{(**)}$	$0.0014X_{ref}$
$n(^{235}U)/n(^{238}U)^{(***)}$	$0.0014X_{ref}$
$n(^{238}Pu)/n(^{239}Pu)^{(****)}$	$0.009X_{ref}$
$n(^{240}Pu)/n(^{239}Pu)^{(****)}$	$0.00055X_{ref}$
$n(^{241}Pu)/n(^{239}Pu)^{(****)}$	$0.0014X_{ref}$
$n(^{242}Pu)/n(^{239}Pu)^{(****)}$	$0.0018X_{ref}$

$^{(*)}$ ITV2010 for Uranium/Plutonium Element Concentration Measurements, applying IDMS for all materials typically encountered in the nuclear fuel cycle under conditions of sufficiently different isotopic compositions of spike and sample and near optimum sample to-spike ratio, using large size spikes (such as LSD) for glove box conditions.

$^{(**)}$ ITV2010 for Uranium/Plutonium Element Concentration Measurements applying IDMS for all materials typically encountered in the nuclear fuel cycle under conditions of sufficiently different isotopic compositions of spike and sample and near optimum sample to spike ratio, using small size spikes for glove box conditions or using large size spikes (such as LSD) for hot cell conditions.

$^{(***)}$ ITV2010 for ^{235}U Abundance Measurements applying TIMS U (0.3% < ^{235}U <1%)

$^{(****)}$ ITV2010 for Plutonium Isotope Assay of Pu and U/Pu Materials – high burnup Pu

<u>zeta score</u>

The zeta score provides an indication of whether the estimate of uncertainty is consistent with the laboratory's deviation from the reference value [4]. 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 [13].

<u>acceptable uncertainty</u>

Since the ITV2010 are expressed as relative combined standard uncertainties, a performance assessment criterion for minimum and maximum acceptable uncertainty to complete satisfactory scores that take reported measurement uncertainties into account was applied in REIMEP-17 [14, 15, 16, 17].

For all $\|\mathrm{zeta}\| \le 2$; it is evaluated whether $0 < u_{lab;rel} \le \mathrm{ITV2010}$ where

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

ITV2010 is the respective International Target Value [1] 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 ITV2010. If this was the case then 'YES' was issued, otherwise 'NO'.

Furthermore, the IUPAC International Harmonised Protocol [8] 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 10-27 list the scores per measurand and participant in detail.

Table 6 summarizes the scores per measurand under investigation. To be consistent the REIMEP-17 participants' results for the uranium and plutonium content were evaluated according to both ITV criteria as described in paragraph 8.1. As there are no ITVs defined for the minor uranium isotope amount ratios, there were no z scores issued for $n(^{234}U)/n(^{238}U)$ and $n(^{236}U)/n(^{238}U)$. The total number of participants in REIMEP-17 (with and without a score) is nine. The participants were requested to measure the measurands specified using their routine analytical procedures. Therefore only the respective participating laboratory has the knowledge, which of the two ITV2010 to apply for evaluating its measurement performance for uranium and plutonium amount content. This is not known to the REIMEP-17 organisers, but it could be assumed already during the design and planning of REIMEP-17 that only a minority of participants would apply IDMS using large size spikes (such as LSD) for glove box conditions. These laboratories are quite well known in the nuclear measurement community. In order not to lower the confidentiality on identity of participants in REIMEP-17 the REIMEP-17 organisers did not ask this information in the questionnaire, but instead decided to calculate for all participants in REIMEP-17 z scores, both for $\hat{\sigma}$ = $0.0009X_{ref}$ and $\hat{\sigma}$ = $0.0014X_{ref}$. Saying this, 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 [8].
Taking into account that the majority of participants did not use large size spikes and thus $\hat{\sigma}$ = $0.0014X_{ref}$**, it can be concluded that the participants in REIMEP-17 performed reasonably well and in compliance with the respective ITV2010. In particular, the measurement performance for the isotope amount ratios is very satisfactory in REIMEP-17 for both samples. This confirms that the ITV2010 are achievable target values under state-of-practice conditions. As can be seen from Table 6 there is room for improvement in reporting uncertainties because for some of the measurands less than 50% of the REIMEP-17 participants with $\|\mathrm{zeta}\| \leq 2$ reported acceptable uncertainties.

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

REIMEP-17A	z score				zeta score				acceptable uncertainty for $\|\mathrm{zeta}\| \leq 2$	z and zeta scores and uncertainty
	S	Q	U	n	S	Q	U	n	YES	S
U(*)	22%	44%	33%	9	56%	11%	33%	9	40%	0%
Pu(*)	22%	56%	22%	9	44%	33%	22%	9	0%	0%
U(**)	78%	11%	11%	9	-	-	-	-	80%	44%
Pu(**)	78%	22%	-	9	-	-	-	-	75%	22%
$n(^{234}U)/n(^{238}U)$	-	-	-	-	56%	22%	22%	9	-	-
$n(^{235}U)/n(^{238}U)$	78%	11%	11%		89%	11%	-	9	75%	67%
$n(^{236}U)/n(^{238}U)$	-	-	-	-	50%	17%	33%	6	-	-
$n(^{238}Pu)/n(^{239}Pu)$	78%	11%	11%	9	78%	11%	11%	9	57%	44%
$n(^{240}Pu)/n(^{239}Pu)$	100%	-	-	9	89%	11%	-	9	88%	78%
$n(^{241}Pu)/n(^{239}Pu)$	89%	11%	-	9	100%	-	-	9	78%	67%
$n(^{242}Pu)/n(^{239}Pu)$	100%	-	-	9	78%	11%	11%	9	71%	56%

(*) Uranium/Plutonium Element Concentration Measurements applying IDMS for all materials typically encountered in the nuclear fuel cycle under conditions of sufficiently different isotopic compositions of spike and sample and near optimum sample-to-spike ratio, using large size spikes (such as LSD) for glove box conditions.

(**) Uranium/Plutonium Element Concentration Measurements applying IDMS for all materials typically encountered in the nuclear fuel cycle under conditions of sufficiently different isotopic compositions of spike and sample and near optimum sample-to-spike ratio, using small size spikes for glove box conditions or using large size spikes (such as LSD) for hot cell conditions.

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

REIMEP-17B	z score				zeta score				acceptable uncertainty for $\|zeta\| \leq 2$	z and zeta scores and uncertainty
	S	Q	U	n	S	Q	U	n	YES	S
U(*)	33%	50%	17%	6	83%	-	17%	6	40%	17%
Pu(*)	33%	-	67%	6	67%	17%	17%	6	25%	17%
U(**)	83%	-	17%	6	-	-	-	6	40%	33%
Pu(**)	33%	17%	50%	6	-	-	-	6	50%	33%
$n(^{234}U)/n(^{238}U)$	-	-	-	-	86%	-	14%	7	-	-
$n(^{235}U)/n(^{238}U)$	86%	14%	-	7	100%	-	-	7	86%	86%
$n(^{236}U)/n(^{238}U)$	-	-	-	-	75%	-	25%	4	-	-
$n(^{238}Pu)/n(^{239}Pu)$	57%	14%	29%	7	71%	-	29%	7	40%	29%
$n(^{240}Pu)/n(^{239}Pu)$	86%	14%	-	7	86%	-	14%	7	67%	57%
$n(^{241}Pu)/n(^{239}Pu)$	57%	43%	-	7	57%	14%	29%	7	25%	14%
$n(^{242}Pu)/n(^{239}Pu)$	86%	14%	-	7	86%	-	14%	7	50%	43%

(*) Uranium/Plutonium Element Concentration Measurements applying IDMS for all materials typically encountered in the nuclear fuel cycle under conditions of sufficiently different isotopic compositions of spike and sample and near optimum sample-to-spike ratio, using large size spikes (such as LSD) for glove box conditions.

(**) Uranium/Plutonium Element Concentration Measurements applying IDMS for all materials typically encountered in the nuclear fuel cycle under conditions of sufficiently different isotopic compositions of spike and sample and near optimum sample-to-spike ratio, using small size spikes for glove box conditions or using large size spikes (such as LSD) for hot cell conditions.

9. Further information extracted from the results

In addition to submission of the results, the participants were asked to answer questions related to their 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

All the participants applied Isotope Dilution Mass Spectrometry (IDMS) for the determination of plutonium and uranium amount content in REIMEP-17. One participant also used K-edge/X-ray fluorescence (XRF). For the measurement of the uranium and plutonium isotope amount ratios, 7 participants applied Thermal Ionisation Mass Spectrometry (TIMS) and 2 participants applied Inductively Coupled Plasma Mass Spectrometry (ICP-MS). 6 laboratories (67%) applied an external correction for mass fractionation using a standard or reference material. In one case the total evaporation techniques was applied. 5 participants used alpha spectrometry for the determination of the $n(^{238}Pu)/n(^{239}Pu)$ amount ratio. A chemical separation prior to measurements was carried out by all the participants.

9.2. A representative study

7 of the 9 participants indicated that the REIMEP-17 sample was treated according to the same analytical procedure routinely used in their laboratory. 8 participants reported that they are experienced in this type of measurement. 2 participants indicated that they analyse 50-100 samples per year, while 7 participants analyse more than 100 samples per year. The majority of the laboratories (89%) are certified, accredited or authorised for this type of analysis. The mission of the laboratories participating in REIMEP-17 is to carry out measurements for fissile material control or safeguards. Some laboratories are also from the field of research & development and medical application. The majority of laboratories routinely analyse samples from reprocessing facilities, safeguards samples and various reference materials samples. 3 laboratories also reported that they analyse other types of samples such as fresh, spent or experimental fuel.

9.3. Quality system and use of standards

All laboratories reported that they are working according to a quality management system; 5 participants according to ISO 17025 and 4 according to ISO 9000 series [18]. All the participants confirmed the participation in various inter-laboratory comparisons. The ILC schemes mentioned were REIMEP, EQRAIN, SME, and ILCs organised by the IAEA and the JAEA. All the participants routinely use certified reference materials mostly for instrument calibration and for method validation. One participant reported using certified reference materials for quality control. The certified reference materials used by the REIMEP-17 participants are given in Annex 28.

9.4. Determination of measurement uncertainty

67% of the participants stated that they routinely report uncertainties on chemical measurements to their customers. 56% of the participants reported uncertainties in REIMEP-17 according to the Guide for Quantifying Measurement Uncertainty (GUM) issued by the International Organization for Standardization (ISO, 2005) and/or EURACHEM/CITAC (2000) [13, 19]. The other participants estimated their measurement uncertainty either via analysis of standards, replicate measurements or by analysing quality control samples. One participant estimated the uncertainty from the ITV2010.

10. Feedback

One participant reported problems with a browser while reporting the results on line. Further it was suggested having a possibility to report the results as scientific notation in order to avoid problems with decimal separators. Another participant stated that the amounts of plutonium and uranium in REIMEP-17B samples were too low to be treated as a routine nuclear material sample. It was also pointed out that there was no information provided with the received samples regarding expected isotopic composition and amount of elements. A drawback in the organisation of REIMEP-17, were the difficulties encountered with transport containers and issues related to the transport of nuclear material.

10.1. Outlook on future REIMEP ILCs

All the participants expressed interest in future REIMEP ILCs. Some participants expressed that they would be interested in samples similar to REIMEP-17. Most of the participants expressed the need for uranium, plutonium or U/Pu mixtures. One participant expressed interest in MOX samples, irradiated and unirradiated fuel samples. Another participant expressed interest in uranium samples containing impurities. Among the elements, plutonium and uranium were mentioned; however some participants would also be interested in neptunium, americium, curium and neodymium. The wish-list concerning the isotopic composition of the samples ranged from depleted uranium (DU) via low enriched uranium (LEU) to high enriched uranium (HEU) and high burn-up plutonium samples.

11. Conclusion

European and international safeguards fulfil an obligation to verify the correctness and completeness of State declarations so that there is credible assurance of the non-diversion of nuclear material from declared activities and of the absence of undeclared nuclear activities. Integral to this process is the laboratory analysis of samples of material collected by safeguards inspectors. Measurement results for nuclear material accountancy and safeguards verification purposes have to be reliable and truly comparable, thus with demonstrated uncertainty and traceability, fit for intended purpose and within the required measurement uncertainties of the ITV2010 [1]. Strict quality control is applied to ensure confidence in the measurement results. The provision of quality control tools for conformity assessment thus directly contributes to the effectiveness of nuclear safeguards systems. One of the IAEA's key objectives is the expansion of the IAEA NWAL for nuclear material analysis. Part of the qualification of candidate laboratories to the NWAL is the participation in inter-laboratory comparisons. In REIMEP-17 two sample solutions with different U and Pu amount contents were prepared to accommodate laboratories with different objectives. One sample was representative for an undiluted input solution; the other sample was a diluted fraction thereof. It can be concluded that the participants in REIMEP-17 performed well for the measurements of uranium and plutonium amount content in compliance with the respective ITV2010 for uranium/plutonium element concentration measurements applying IDMS for all materials typically encountered in the nuclear fuel cycle under conditions of sufficiently different isotopic compositions of spike and sample and near optimum sample to spike ratio, using small size spikes for glove box conditions or using large size spikes (such as LSD) for hot cell conditions for a synthetic input solution sample. In particular, the measurement performance for the isotope amount ratios was very satisfactory for both REIMEP-17 samples. This confirms the measurement capabilities of laboratories in the field of nuclear material analysis and at the same time serves as a confirmation that the stringent ITV2010 are achievable target values under state-of-practice conditions. As expected, the spread of results was larger for the minor uranium isotope amount ratios; also for some of the measurands differences in the uncertainty estimates provided by laboratories were observed, even when using the same instrumental technique.

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Abbreviations

ANOVA	Analysis of Variance
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
DU	Depleted Uranium
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
HEU	High enriched uranium
IAEA	International Atomic Energy Agency
ICP-MS	Inductively Coupled Plasma Mass Spectrometry
ILC	Inter-laboratory Comparison
IMEP	Inter-laboratory Measurement Evaluation Programme
JRC-IRMM	Institute for Reference Materials and Measurements, JRC, European Commission
ID-TIMS	Isotope Dilution Thermal Ionisation Mass Spectrometry
ISO	International Organization for Standardization
JRC-ITU	Institute for Transuranium Elements, JRC, European Commission
ITV	International Target Value
IUPAC	International Union for Pure and Applied Chemistry
JAEA	Japan Atomic Energy Agency
JRC	Joint Research Centre, European Commission
LEU	Low enriched uranium
MOX	Mixed oxide
NBL	New Brunswick Laboratory
NPT	Treaty on the Non-Proliferation of Nuclear Weapons
NU	Natural Uranium
NWAL	Network of Analytical Laboratories
REIMEP	Regular European Inter-laboratory Measurement Evaluation Programme
SME	Safeguards Measurement Evaluation (NBL)
TIMS	Thermal Ionisation Mass Spectrometry

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Annexes

Annex 1: Results of the homogeneity assessment in REIMEP-17A

REIMEP-17A	Pu content [µmol/g]			U content [µmol/g]		
Ampoule ID	Rep 1	Rep 2	Rep 3	Rep 1	Rep 2	Rep 3
34	9.15858	9.15717	9.15525	843.500	843.302	843.304
48	9.15581	9.15357	9.15677	843.524	843.493	843.661
19	9.15862	9.15667	9.16012	843.498	843.666	843.561
21	9.15595	9.15159	9.15327	842.753	843.132	843.787
5	9.15667	9.15619	9.15599	843.476	843.244	/
mean	9.15615			843.422		
$\hat{\sigma}_{rel}$ [%]	0.09			0.09		
0.3* $\hat{\sigma}_{rel}$ [%]	0.027			0.027		
$s_{bb,\ rel}$ [%]	0.017			MSB<MSW		
$s_{wb,\ rel}$ [%]	0.018			0.031		
$u_{bb,\ rel}$ [%]	0.007			0.013		
$s_{bb,\ rel}$ ($u_{bb,\ rel}$) < 0.3* $\hat{\sigma}_{rel}$	YES			YES		

REIMEP-17A	$n(^{238}Pu)/n(^{239}Pu)$			$n(^{240}Pu)/n(^{239}Pu)$		
Ampoule ID	Rep 1	Rep 2	Rep 3	Rep 1	Rep 2	Rep 3
34	0.04196	0.04196	0.04196	0.47866	0.47867	0.4787
48	0.04195	0.04194	0.04195	0.47867	0.47869	0.47865
19	0.04192	0.04191	0.04192	0.47863	0.47865	0.47862
21	0.04196	0.04195	0.04197	0.47866	0.47871	0.4787
5	0.04194	0.04194	0.04194	0.47868	0.47868	0.47869
mean	0.041945			0.47867		
$\hat{\sigma}_{rel}$ [%]	0.90			0.055		
0.3* $\hat{\sigma}_{rel}$ [%]	0.27			0.017		
$s_{bb,\ rel}$ [%]	0.042			0.004		
$s_{wb,\ rel}$ [%]	0.014			0.004		
$u_{bb,\ rel}$ [%]	0.005			0.002		
$s_{bb,\ rel}$ ($u_{bb,\ rel}$) < 0.3* $\hat{\sigma}_{rel}$	YES			YES		

REIMEP-17A	$n(^{241}Pu)/n(^{239}Pu)$			$n(^{242}Pu)/n(^{239}Pu)$		
Ampoule ID	Rep 1	Rep 2	Rep 3	Rep 1	Rep 2	Rep 3
34	0.12405	0.12408	0.12409	0.13774	0.13774	0.13774
48	0.12401	0.12402	0.12401	0.13774	0.13774	0.13774
19	0.12382	0.12381	0.12380	0.13775	0.13776	0.13775
21	0.12406	0.12410	0.12409	0.13774	0.13774	0.13774
5	0.12396	0.12397	0.12398	0.13775	0.13775	0.13775
mean	0.12399			0.137745		
$\hat{\sigma}_{rel}$ [%]	0.14			0.18		
0.3* $\hat{\sigma}_{rel}$ [%]	0.042			0.054		
$s_{bb, rel}$ [%]	0.089			0.005		
$s_{wb, rel}$ [%]	0.012			0.002		
$u_{bb, rel}$ [%]	0.005			0.001		
$s_{bb, rel}$ ($u_{bb, rel}$) < 0.3* $\hat{\sigma}_{rel}$	NO			YES		

REIMEP-17A	$n(^{234}U)/n(^{238}U)$			$n(^{235}U)/n(^{238}U)$		
Ampoule ID	Rep 1	Rep 2	Rep 3	Rep 1	Rep 2	Rep 3
34	0.0000706	0.0000715	0.0000707	0.0068099	0.0068092	0.0068096
48	0.0000726	0.0000708	0.0000711	0.0068104	0.0068117	0.0068091
19	0.0000721	0.0000702	0.0000725	0.0068055	0.0068082	0.0068064
21	0.0000703	0.0000719	0.0000725	0.0068060	0.0068111	0.0068080
5	0.0000703	0.0000735	/	0.0068153	0.006813	/
mean	0.0000715			0.0068095		
$\hat{\sigma}_{rel}$ [%]	3.76			0.14		
0.3* $\hat{\sigma}_{rel}$ [%]	1.13			0.042		
$s_{bb, rel}$ [%]	MSB<MSW			0.035		
$s_{wb, rel}$ [%]	1.69			0.024		
$u_{bb, rel}$ [%]	0.69			0.010		
$s_{bb, rel}$ ($u_{bb, rel}$) < 0.3* $\hat{\sigma}_{rel}$	YES			YES		

REIMEP-17A	$n(^{236}U)/n(^{238}U)$		
Ampoule ID	Rep 1	Rep 2	Rep 3
34	0.0000061	0.0000042	0.0000056
48	0.000003	0.0000065	0.000004
19	0.0000037	0.0000042	0.0000045
21	0.0000055	0.0000057	0.0000036
5	0.0000071	0.0000055	/
mean	0.00000494		
$\hat{\sigma}_{rel}$ [%]	44		
0.3* $\hat{\sigma}_{rel}$ [%]	13.2		
$s_{bb, rel}$ [%]	5.9		
$s_{wb, rel}$ [%]	24.1		
$u_{bb, rel}$ [%]	9.9		
$s_{bb, rel}$ ($u_{bb, rel}$)< 0.3* $\hat{\sigma}_{rel}$	YES		

Annex 2: Results of the homogeneity assessment in REIMEP-17B

REIMEP-17B	Pu content [nmol/g]			U content [µmol/g]		
Ampoule ID	Rep 1	Rep 2	Rep 3	Rep 1	Rep 2	Rep 3
43	22.9817	22.9800	22.9784	2.11718	2.11825	2.11723
53	22.9798	22.9772	22.9729	2.11645	2.11717	2.11648
12	22.9807	22.9775	22.9748	2.11678	2.11732	2.11766
21	22.9769	22.9785	/	2.11781	2.11667	2.11671
5	22.9685	22.9659	22.9718	2.11453	2.11553	/
mean	22.9760			2.11684		
$\hat{\sigma}_{rel}$ [%]	0.09			0.09		
0.3* $\hat{\sigma}_{rel}$ [%]	0.027			0.027		
$s_{\mathrm{bb,\ rel}}$ [%]	0.018			0.039		
$s_{\mathrm{wb,\ rel}}$ [%]	0.012			0.026		
$u_{\mathrm{bb,\ rel}}$ [%]	0.005			0.011		
$s_{\mathrm{bb,\ rel}}$ ($u_{\mathrm{bb,\ rel}}$) < 0.3* $\hat{\sigma}_{rel}$	YES			NO		

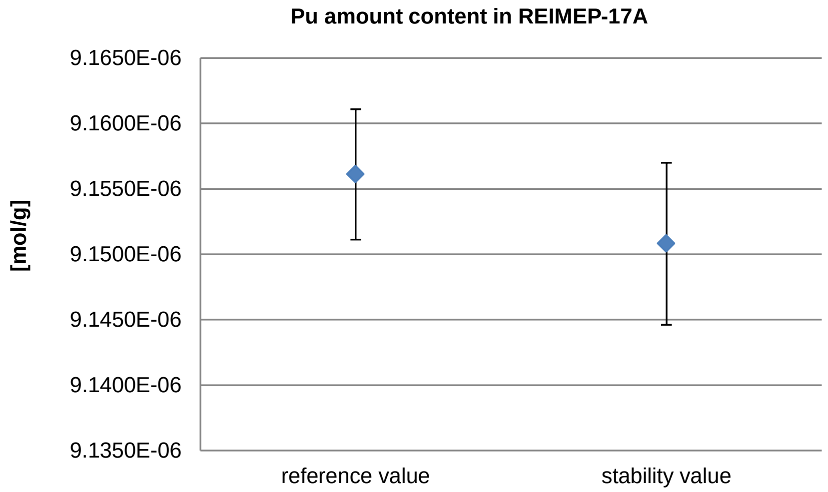
REIMEP-17B	$n(^{238}\mathrm{Pu})/n(^{239}\mathrm{Pu})$			$n(^{240}\mathrm{Pu})/n(^{239}\mathrm{Pu})$		
Ampoule ID	Rep 1	Rep 2	Rep 3	Rep 1	Rep 2	Rep 3
43	0.03962	0.03962	0.03962	0.47844	0.47846	0.47846
53	0.03906	0.03904	0.03902	0.47839	0.47841	0.47841
12	0.03875	0.03875	0.03872	0.47841	0.47838	0.47839
21	0.03927	0.03924	/	0.47843	0.47843	/
5	0.03897	0.03895	0.03897	0.47842	0.4784	0.47842
mean	0.03911			0.47842		
$\hat{\sigma}_{rel}$ [%]	0.9			0.055		
0.3* $\hat{\sigma}_{rel}$ [%]	0.27			0.017		
$s_{\mathrm{bb,\ rel}}$ [%]	0.88			0.005		
$s_{\mathrm{wb,\ rel}}$ [%]	0.039			0.002		
$u_{\mathrm{bb,\ rel}}$ [%]	0.016			0.001		
$s_{\mathrm{bb,\ rel}}$ ($u_{\mathrm{bb,\ rel}}$) < 0.3* $\hat{\sigma}_{rel}$	NO			YES		

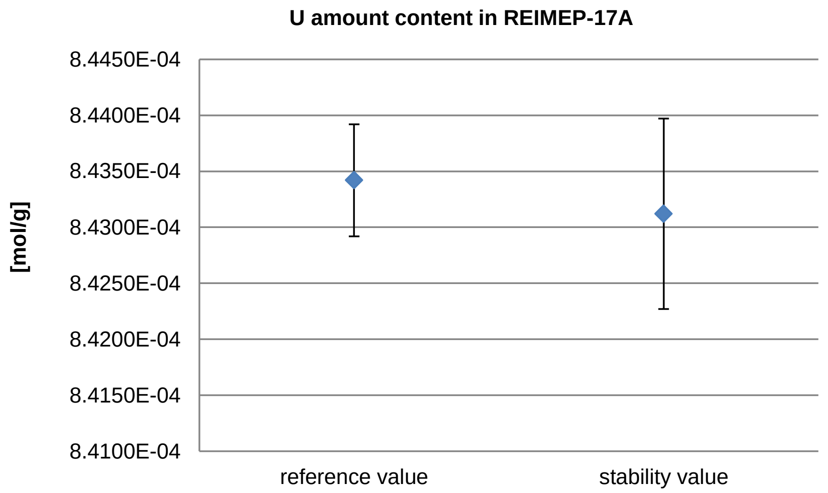
REIMEP-17B	$n(^{241}Pu)/n(^{239}Pu)$			$n(^{242}Pu)/n(^{239}Pu)$		
Ampoule ID	Rep 1	Rep 2	Rep 3	Rep 1	Rep 2	Rep 3
43	0.11322	0.11325	0.11325	0.138623	0.138623	0.138623
53	0.11063	0.11065	0.11062	0.138841	0.138841	0.138841
12	0.1092	0.10921	0.10922	0.138959	0.138959	0.138959
21	0.11164	0.111161	/	0.138757	/	0.138757
5	0.11027	0.11028	0.11026	0.138870	0.138871	0.138870
mean	0.11092			0.138814		
$\hat{\sigma}_{rel}$ [%]	0.14			0.18		
0.3* $\hat{\sigma}_{rel}$ [%]	0.04			0.054		
$s_{bb,\,rel}$ [%]	1.4			0.094		
$s_{wb,\,rel}$ [%]	0.10			0.0001		
$u_{bb,\,rel}$ [%]	0.04			0.0001		
$s_{bb,\,rel}$ ($u_{bb,\,rel}$) < 0.3* $\hat{\sigma}_{rel}$	NO			NO		

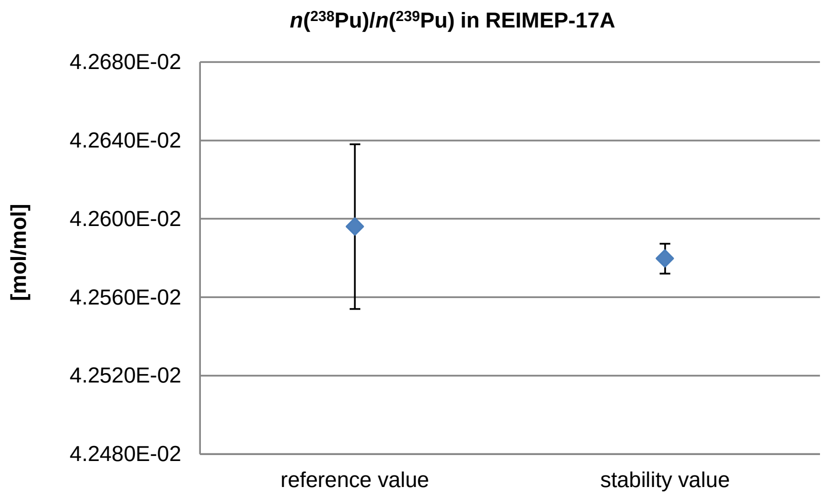
REIMEP-17B	$n(^{234}U)/n(^{238}U)$			$n(^{235}U)/n(^{238}U)$		
Ampoule ID	Rep 1	Rep 2	Rep 3	Rep 1	Rep 2	Rep 3
43	0.0000887	0.0000874	0.0000865	0.0068098	0.006814	0.0068127
53	0.0000932	0.0000951	0.0000939	0.0068145	0.0068197	0.0068128
12	0.0000935	0.0000953	0.0000933	0.006814	0.0068091	0.0068098
21	0.0000898	0.000091	0.000091	0.0068115	0.0068109	0.0068144
5	0.0000925	0.0000891	/	0.0068096	0.0068098	/
mean	0.0000915			0.00681		
$\hat{\sigma}_{rel}$ [%]	6.45			0.14		
0.3* $\hat{\sigma}_{rel}$ [%]	1.94			0.042		
$s_{bb,\,rel}$ [%]	3.0			0.023		
$s_{wb,\,rel}$ [%]	1.34			0.04		
$u_{bb,\,rel}$ [%]	0.55			0.015		
$s_{bb,\,rel}$ ($u_{bb,\,rel}$) < 0.3* $\hat{\sigma}_{rel}$	NO			YES		

REIMEP-17B	$n(^{236}U)/n(^{238}U)$		
Ampoule ID	Rep 1	Rep 2	Rep 3
43	0.0000101	0.0000072	0.0000048
53	0.000006	0.0000043	0.0000068
12	0.0000083	0.0000117	0.0000067
21	0.0000087	0.0000085	0.0000057
5	0.0000073	0.000006	/
mean		0.0000073	
$\hat{\sigma}_{rel}$ [%]		25	
0.3* $\hat{\sigma}_{rel}$ [%]		7.5	
$s_{bb, rel}$ [%]		1.7	
$s_{wb, rel}$ [%]		28	
$u_{bb, rel}$ [%]		11	
$s_{bb, rel}$ ($u_{bb, rel}$)< 0.3* $\hat{\sigma}_{rel}$		NO	

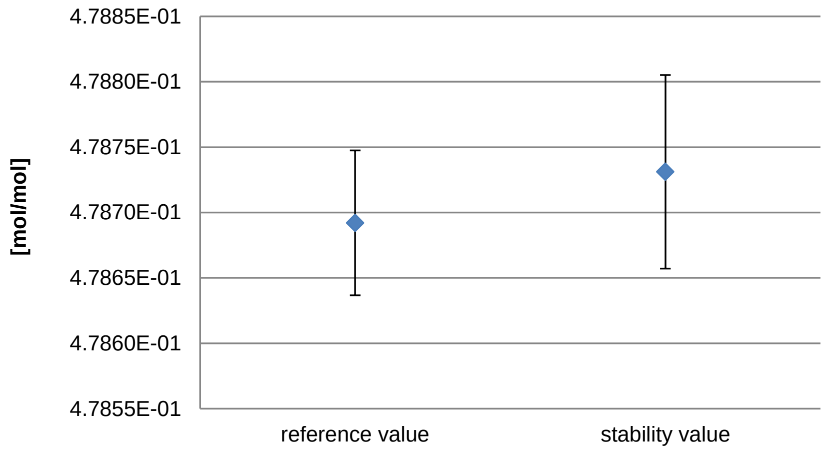
Annex 3: Results of the stability assessment in REIMEP-17A



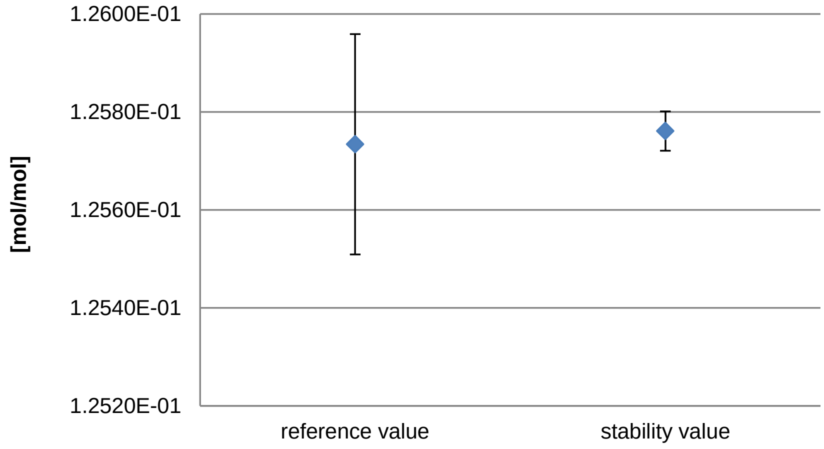




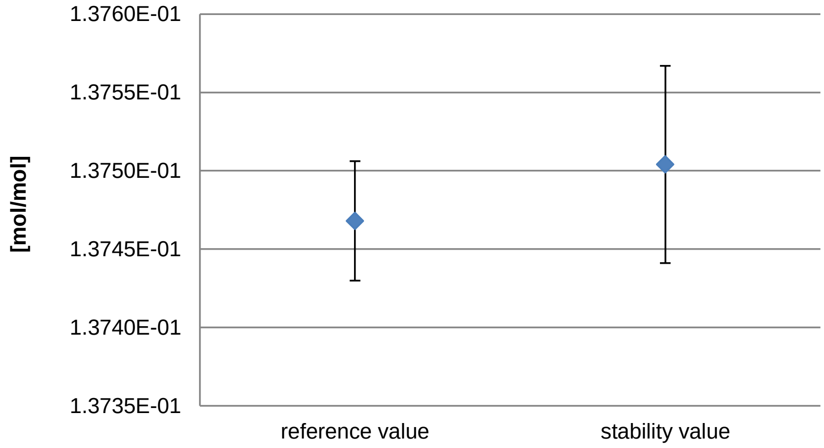
$n(^{240}Pu)/n(^{239}Pu)$ in REIMEP-17A



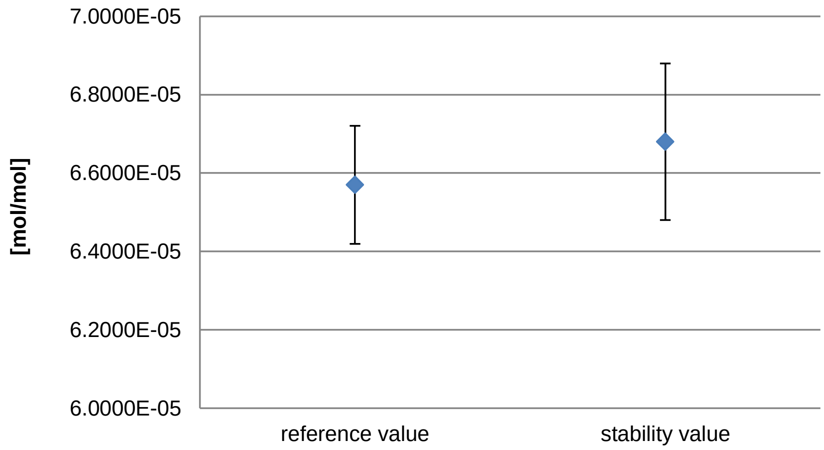
$n(^{241}Pu)/n(^{239}Pu)$ in REIMEP-17A



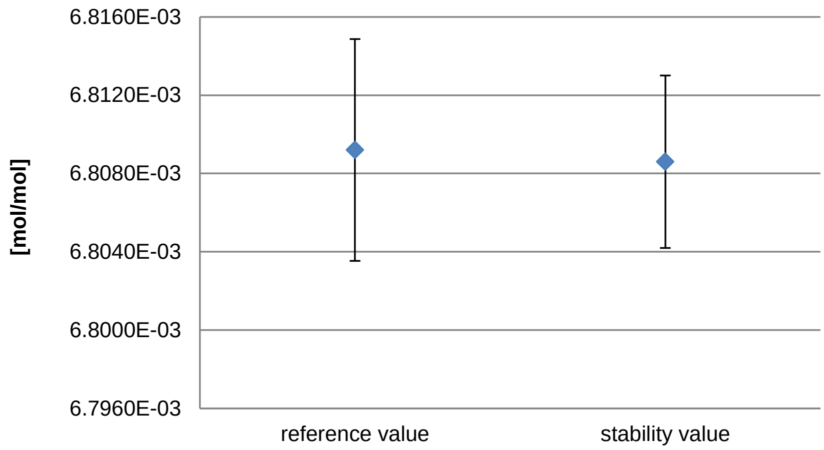
$n(^{242}Pu)/n(^{239}Pu)$ in REIMEP-17A



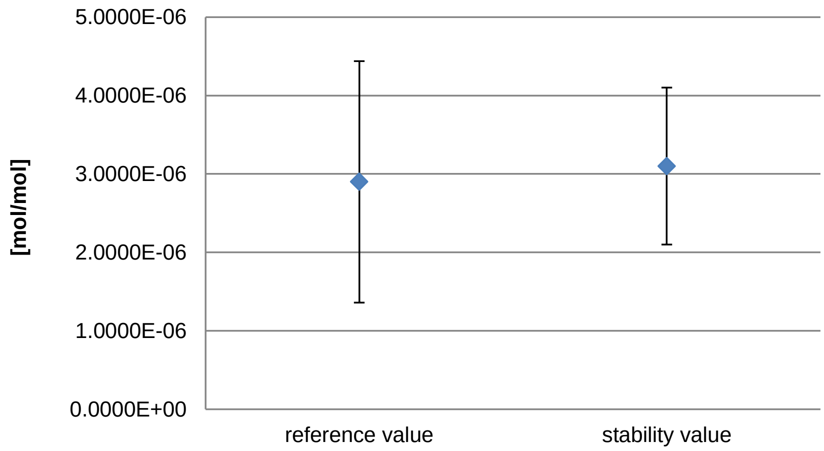
$n(^{234}U)/n(^{238}U)$ in REIMEP-17A



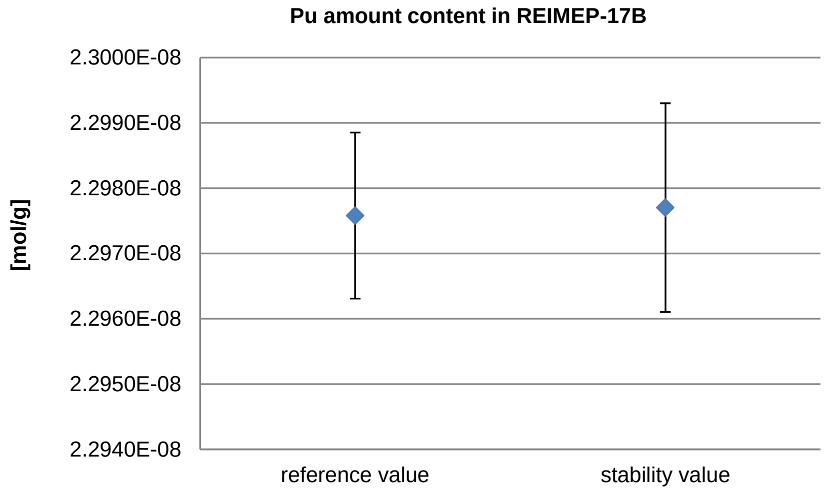
$n(^{235}U)/n(^{238}U)$ in REIMEP-17A

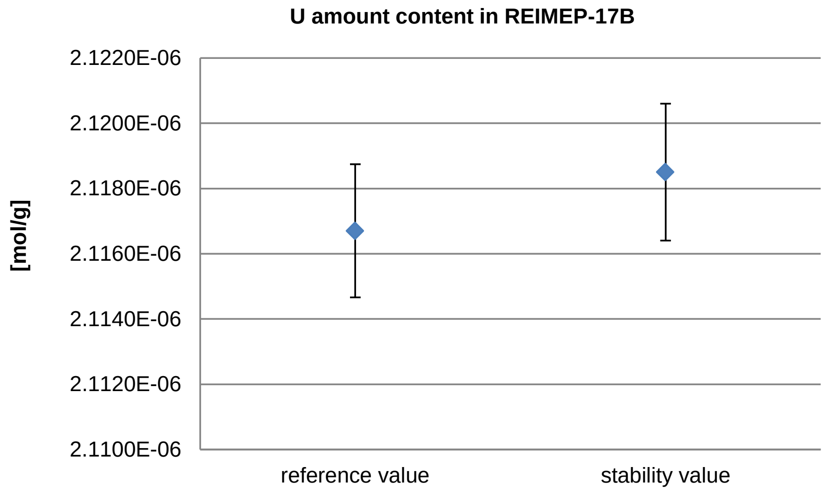


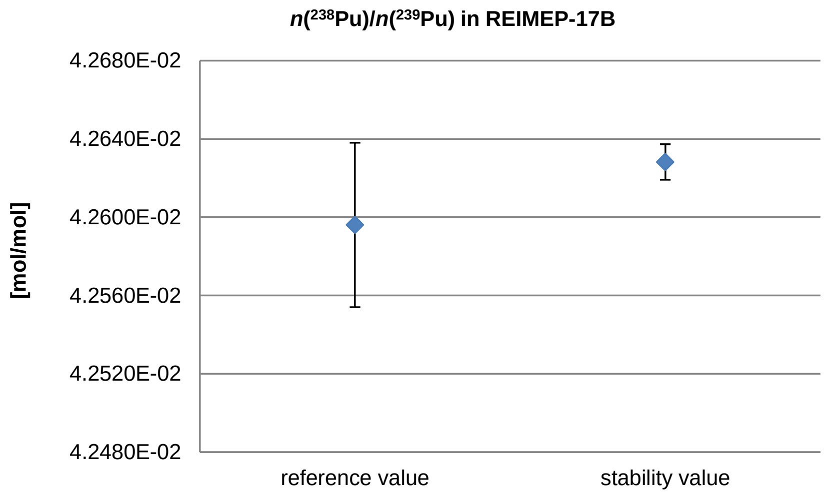
$n(^{236}U)/n(^{238}U)$ in REIMEP-17A

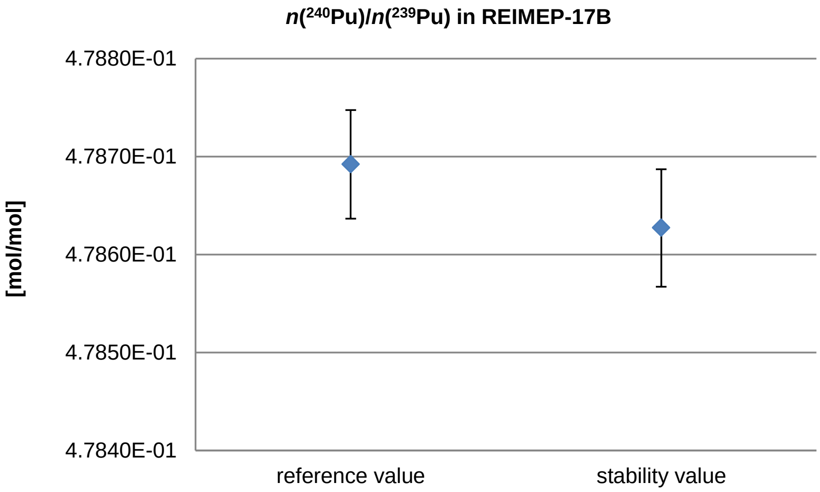


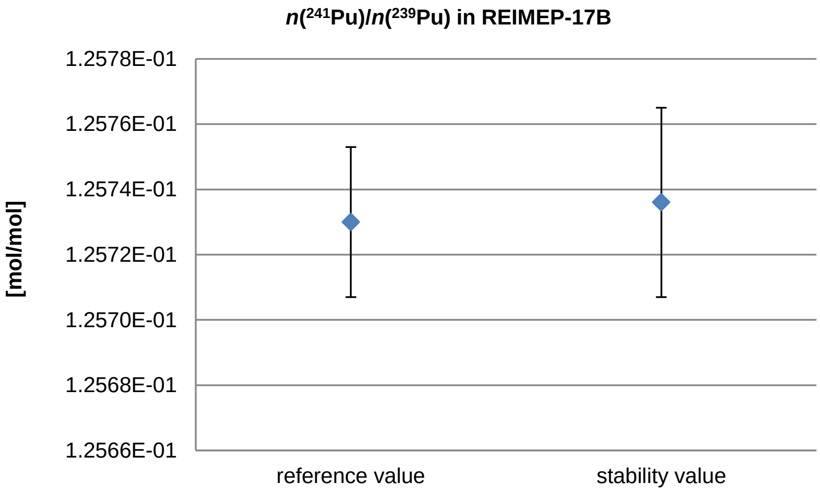
Annex 4: Results of the stability assessment in REIMEP-17B

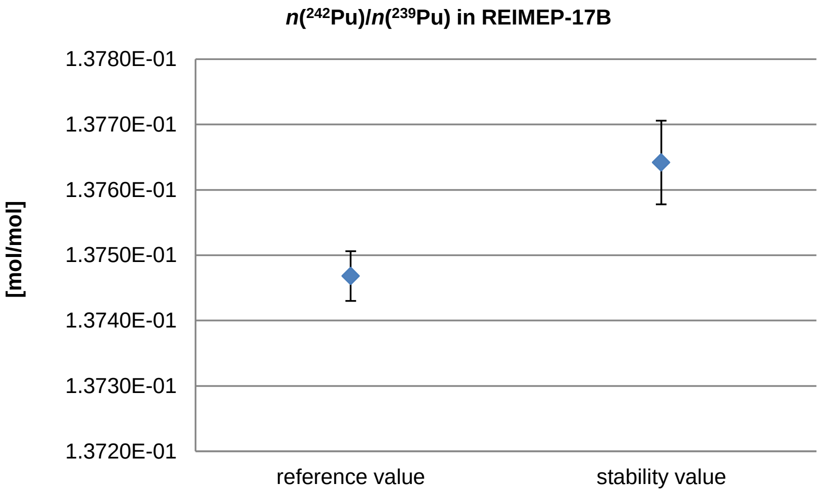


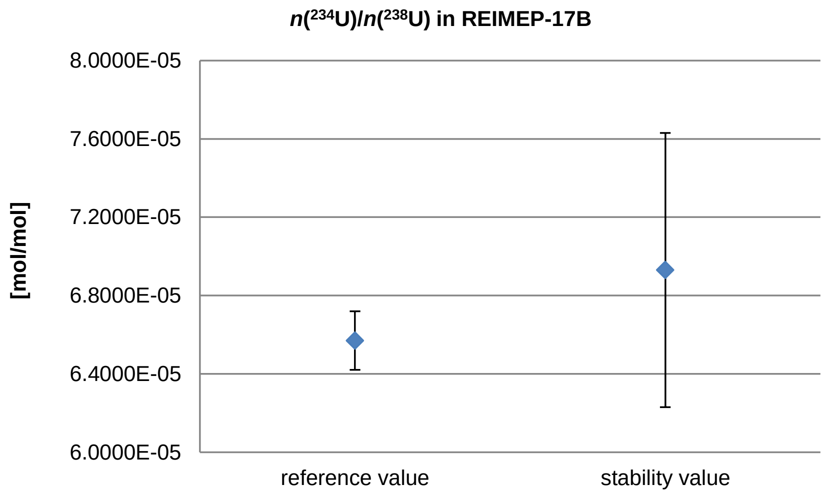




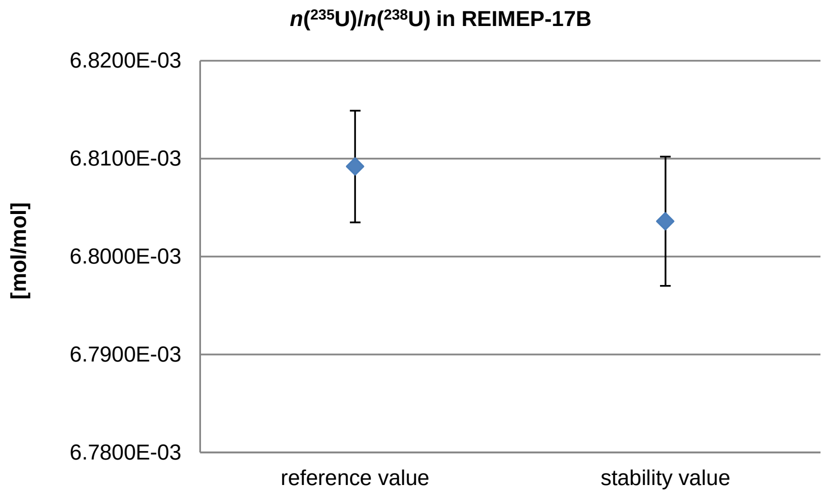

n(240Pu)/n(239Pu) in REIMEP-17B
4.7880E-01
4.7870E-01
4.7860E-01
4.7850E-01
4.7840E-01
[mol/mol]
reference value
stability value


n(241Pu)/n(239Pu) in REIMEP-17B
1.2578E-01
1.2576E-01
1.2574E-01
1.2572E-01
1.2570E-01
1.2568E-01
1.2566E-01
[mol/mol]
reference value
stability value

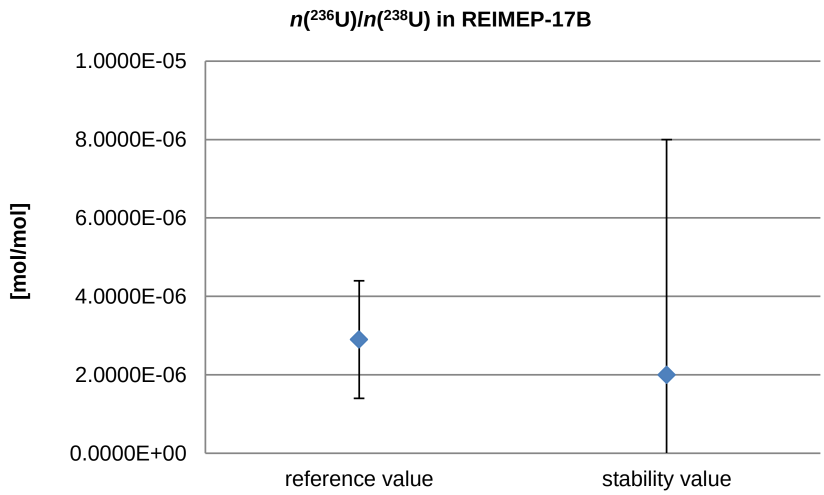

n(242Pu)/n(239Pu) in REIMEP-17B
1.3780E-01
1.3770E-01
1.3760E-01
1.3750E-01
1.3740E-01
1.3730E-01
1.3720E-01
[mol/mol]
reference value
stability value



$n(^{234}U)/n(^{238}U)$ in REIMEP-17B
8.0000E-05
7.6000E-05
7.2000E-05
6.8000E-05
6.4000E-05
6.0000E-05
[mol/mol]
reference value
stability value



$n(^{235}U)/n(^{238}U)$ in REIMEP-17B
6.8200E-03
6.8100E-03
6.8000E-03
6.7900E-03
6.7800E-03
[mol/mol]
reference value
stability value



$n(^{236}U)/n(^{238}U)$ in REIMEP-17B
1.0000E-05
8.0000E-06
6.0000E-06
4.0000E-06
2.0000E-06
0.0000E+00
[mol/mol]
reference value
stability value

Annex 5: Invitation letter

Geel, 30 March 2012

JRC.D.2/RJa/YAr/mt/ARES(2012) 12-046/380201

The IRMM Regular European Interlaboratory Measurement Evaluation Programme

REIMEP-17: Interlaboratory Comparison on plutonium and uranium amount content, and isotope amount ratios in synthetic input solution

The Regular European Interlaboratory Measurement Evaluation Programme (REIMEP) was started by IRMM in 1982 for carrying out external control of the quality of the measurements of the nuclear fuel cycle materials. In REIMEP campaigns, samples matching materials analysed routinely in the nuclear fuel cycle are sent to participating laboratories for measurements and to date 16 REIMEP campaigns have been organised, which involved safeguards laboratories throughout the world.
http://irmm.jrc.ec.europa.eu/interlaboratory_comparisons/reimep

We would like to announce the forthcoming REIMEP-17 interlaboratory comparison: "Plutonium and uranium amount content, and isotope amount ratios in synthetic input solution" and invite laboratories to participate.

Participating laboratories in REIMEP-17 receive two samples with undisclosed U, Pu amount content and 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 $n(^{234}U)/n(^{238}U)$, $n(^{235}U)/n(^{238}U)$, $n(^{236}U)/n(^{238}U)$.

One sample will have a U, Pu amount content typical for undiluted input solution and the other sample is a diluted fraction thereof.

Those isotope amount ratios are to be measured by participating laboratories using their routine analytical procedures. 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.

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 REIMEP-17 samples is exhausted.

The participation fee for REIMEP-17 is **€ 2500,** which includes sample dispatch.

The REIMEP-17 samples will be shipped from the Institute for Transuranium Elements (EC-JRC-Karlsruhe) to the participants. Each participant has to request the Import licence in time to enable shipment. The handling or operation licence will be organized by the ITU Transport officers.

We ask each participant to provide the following information:

1) contact person (e-mail address and telephone number)
2) contact person for nuclear transport licensing
3) contact person for dangerous goods
4) nuclear accountancy area
5) delivery address (not a PO box but a real address)

Institute for Reference Materials and Measurements
Retieseweg 111, 2440 Geel, Belgium
Tel.: +32-(0)14-571 617 • Fax: +32-(0)14-571 863
jrc-irmm-reimep@ec.europa.eu • http://www.irmm.jrc.be

With registering in REIMEP-17 the participant agrees to the *Transfer of Title and Risks*: "Title and risks associated with the samples provided by ITU shall pass to the participants upon delivery of the samples to their premises". Participants will also be responsible for the sample disposal and costs involved.

Please register electronically for this interlaboratory comparison using the following link: https://web.jrc.ec.europa.eu/ilcRegistrationWeb/registration/registration.do?selComparison=861

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 new deadline for registration is **15 May 2012**. After the deadline the participants in REIMEP-17 will be contacted by the ITU transport officer regarding shipment and transport. The samples will be sent to participants May-October 2012. 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,

Rožle Jakopič
REIMEP-17 Co-ordinator

Yetunde Aregbe
IRMM Safeguards Co-ordinator

Institute for Reference Materials and Measurements
Retieseweg 111, 2440 Geel, Belgium
Tel.: +32-(0)14-571 617 • Fax: +32-(0)14-571 863
jrc-irmm-reimep@ec.europa.eu • http://www.irmm.jrc.be

Annex 6: Confirmation of registration

JAKOPIC Rozle (JRC-GEEL)

From:	JAKOPIC Rozle (JRC-GEEL) on behalf of JRC IRMM REIMEP
Sent:	24 April 2012 08:57
To:	
Subject:	

Dear

Thank you for registering in REIMEP-17. A separate e-mail will follow shortly with a laboratory code for reporting results.

Should you have any additional question do not hesitate to contact me.

Nice regards,
Rozle

Dr. Rožle JAKOPIČ
Laboratory Responsible Nuclear Chemistry

European Commission
Joint Research Centre (JRC)
Unit D.2 Reference Materials

Institute for Reference Materials and Measurements
Retieseweg 111, B-2440 Geel
tel: +32 14 571 617
fax: +32 14 571 548
e-mail: rozle.jakopic@ec.europa.eu

The views expressed are purely those of the writer and may not in any circumstances be regarded as stating an official position of the European Commission

Annex 7: Accompanying letter

Geel, 13 June 2012
ARES number

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

REIMEP-17: Interlaboratory Comparison on plutonium and uranium amount content, and isotope amount ratios in synthetic input solution

Dear «TITLE» «SURNAME»,

Thank you for your participation in REIMEP-17.
Together with this letter, we are sending you two ampoules of REIMEP-17 solution for analyses with undisclosed U, Pu amount content and isotope amount ratio values as described in the REIMEP-17 announcement. REIMEP-17 A has a U, Pu amount content typical for undiluted input solution and REIMEP-17 B is a diluted fraction thereof:

http://irmm.jrc.ec.europa.eu/interlaboratory_comparisons/reimep/reimep-17/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).

The U, Pu amount content and isotope amount ratios are to be measured in both samples. Participants in NUSIMEP are asked to apply the same measurement procedure as used in routine sample analysis of this kind. Please be aware that the reference date for your measurements results is **1 March 2013!** For more information, please consult the participant's 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)14-571 617 • Fax: +32-(0)14-571 863
jrc-irmm-reimep@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,

Rožle Jakopič
REIMEP-17 Co-ordinator

Yetunde Aregbe
IRMM Safeguards Co-ordinator

Annexes:

- Participants' guidelines for reporting

- Confirmation of sample receipt

- Checklist

Retieseweg 111, 2440 Geel, Belgium
Tel.: +32-(0)14-571 617 • Fax: +32-(0)14-571 863
jrc-irmm-reimep@ec.europa.eu • http://irmm.jrc.ec.europa.eu/

Annex 8: Questionnaire

Milc questionnaire

Comparison for REIMEP-17

Please complete this form together with the result reporting form. All anwers 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) Research and development
- ☐ b) Measurement of radioactivity in the environment
- ☐ c) Monitoring of nuclear facilities
- ☐ d) Medical application
- ☐ e) Measurements for fissile material control or safeguards
- ☐ f) Network of Analytical Laboratories (NWAL)
- ☐ g) 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:

- Page 1 of 7 -

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

- ○ no
- ○ yes

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?**

- ○ a) < 25
- ○ b) 25-50
- ○ c) 50-100
- ○ d) > 100

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

- ○ Experienced
- ○ Less experienced
- ○ Not experienced

7. **On what type of samples do you routinely perform uranium and plutonium assay analysis (you can make more than one choice)?**

- ☐ a) Safeguards samples
- ☐ b) Forensic samples
- ☐ c) Samples from reprocessing facilities
- ☐ d) Reference materials
- ☐ e) Other

7.1. If Other, please specify:

[ ]

8. **How did determine the uranium content in REIMEP-17 samples?**

- ☐ a) Isotope Dilution Mass Spectrometry (IDMS)
- ☐ b) Davies and Gray titration
- ☐ c) K-edge densitometry
- ☐ d) X-Ray Fluorescence (XRF)
- ☐ e) Other

8.1. If other, please specify:

9. **How did you determine the plutonium content in REIMEP-17 samples?**

- [ ] a) Isotope Dilution Mass Spectrometry (IDMS)
- [ ] b) Coulometric titration
- [ ] c) K-edge densitometry
- [ ] d) X-Ray Fluorescence (XRF)
- [ ] e) Other

9.1. If other, please specify:

10. **Was the REIMEP-17 sample treated according to the same analytical procedure routinely used in your laboratory?**

- ○ no
- ○ yes

10.1. If no, please specify why not: *

11. **Did you perform a chemical separation prior to measurement?**

- ○ no
- ○ yes

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

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

- ○ no
- ○ yes

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

- ○ applicable
- ○ not applicable

13.1. If applicable, please choose *

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

13.1.1. If other, please specify *

[ ]

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

- ◯ applicable
- ◯ not applicable

14.1. Equipment *

[ ]

14.2. Detector *

[ ]

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

- ◯ applicable
- ◯ not applicable

15.1. If applicable, please choose: *

- ◯ no
- ◯ yes

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

[ ]

16. 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

16.1. Mass-spectrometer (type) *

[ ]

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

16.3. Any particular experimental parameters *

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

- ○ no
- ○ yes

17.1. If yes, please specify: *

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

- ○ no
- ○ yes

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

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

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

18.2.1. If other, please specify:

19. 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

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

- ○ no
- ○ yes

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

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

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

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

- no
- yes

22. **How did you learn about REIMEP-17 (you can make more than one choice)?**

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

22.1. If other, please specify:

23. **Would you be interested in participating in future REIMEP inter-laboratory comparisons?**

- no
- yes

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

23.2. Which isotope(s) or mixture of isotopes? *

23.3. What type of matrices? *

23.4. What concentration level? *

23.5. Other, please specify: *

24. **Do you have any feedback/comments on REIMEP-17?**

25. **Questionnaire completed by:**

25.1. Name

25.2. Position:

Annex 9: Confirmation of sample receipt

Geel, 13 June 2012
JRC.D.2/RB/mt/ARES(2012) 12-068/ 720789

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

REIMEP-17

Confirmation of receipt of two ampoules of input 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 CODES ……..…………………………-…………….

……..…………………………-…………….

ANY REMARKS ……………………………………………….

……………………………………………….

Date of package arrival ……………………………………………….

Signature ……………………………………………….

Please return this form to:

Rožle Jakopič
REIMEP-17 Co-ordinator
EC-JRC-IRMM
Retieseweg 111
B-2440 GEEL
BELGIUM
Fax : +32 14 571 863
e-mail : jrc-irmm-reimep@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)14 571 617. Fax: +32 (0)14 571 863. E-mail: JRC-IRMM-REIMEP@ec.europa.eu

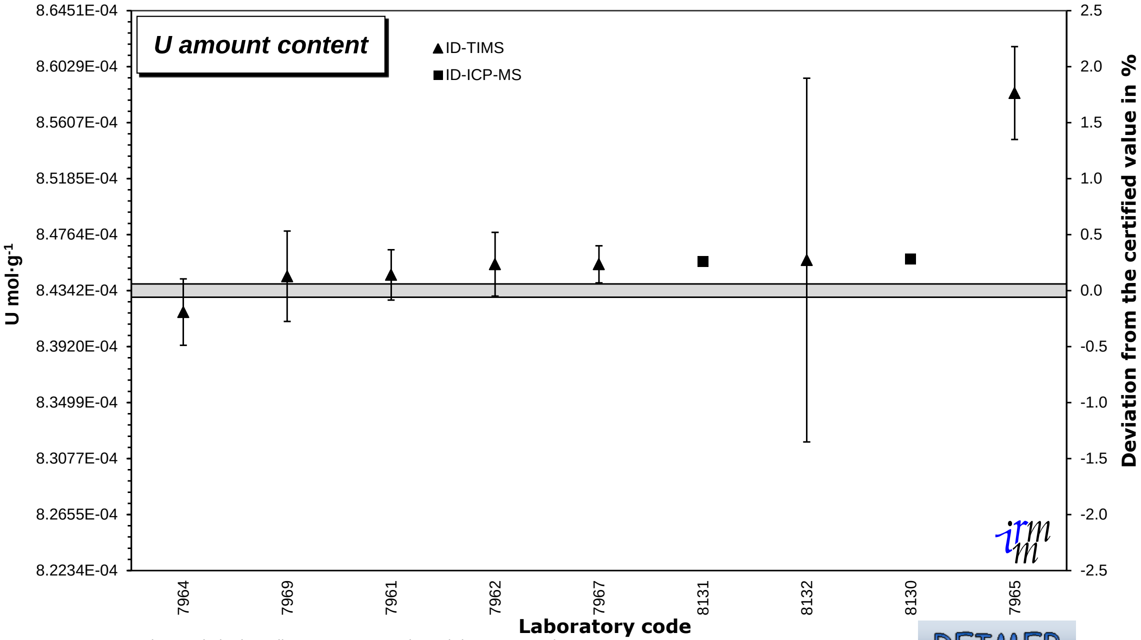
Annex 10: Results for the uranium amount content in REIMEP-17A

Laboratory	Analytical method	Reported uranium content mol·g^{-1}	Reported uncertainty uranium content mol·g^{-1}	Coverage factor k
7961	IDMS	8.4460E-04	1.9000E-06	1
7962	IDMS	8.4540E-04	2.4000E-06	2
7964	IDMS	8.4180E-04	2.5000E-06	2
7965	IDMS	8.5830E-04	3.5000E-06	2
7967	IDMS	8.4540E-04	1.4000E-06	2
7969	IDMS	8.4450E-04	3.4000E-06	2
8130	IDMS	8.4580E-04	2.0000E-07	2
8131	IDMS	8.4560E-04	2.0000E-07	2
8132	IDMS	8.4572E-04	1.3700E-05	1

Laboratory	ITV: 0.18% z score	ITV: 0.28% z score	zeta score	ITV: 0.18% acceptable uncertainty for $\|zeta\| \leq 2$	ITV: 0.28% acceptable uncertainty for $\|zeta\| \leq 2$
7961	1.55	1.00	0.62	NO	YES
7962	2.61	1.68	1.62	YES	YES
7964	-2.13	-1.37	-1.27	YES	YES
7965	19.60	12.60	8.42	-	-
7967	2.61	1.68	2.66	-	-
7969	1.42	0.91	0.63	NO	YES
8130	3.14	2.02	8.84	-	-
8131	2.87	1.85	8.10	-	-
8132	3.03	1.95	0.17	NO	NO

REIMEP-17A: Plutonium and uranium amount content, and isotope amount ratios in synthetic input solution

Certified value for U amount content: 843.42 ± 0.50 µmol/g [$U=k \cdot u_c(k=2)$]



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 ($X_{ref} \pm 2u_{ref}$).

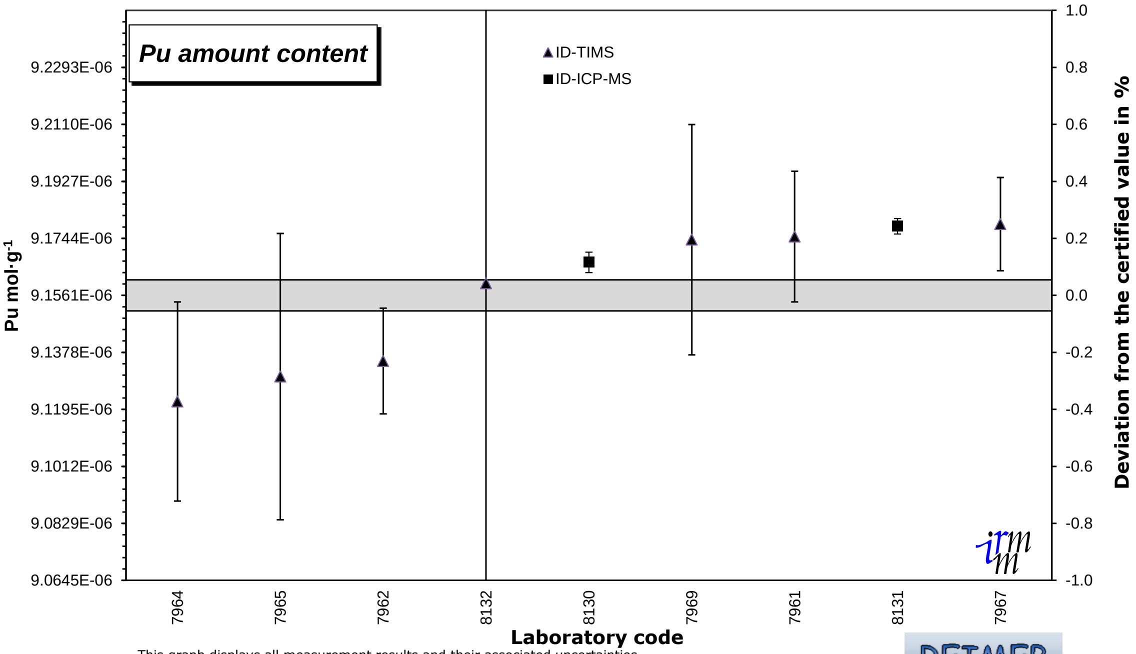
Annex 11: Results for the plutonium amount content in REIMEP-17A

Laboratory	Analytical method	Reported plutonium content *mol·g^-1*	Reported uncertainty plutonium content *mol·g^-1*	Coverage factor *k*
7961	IDMS	9.1750E-06	2.1000E-08	1
7962	IDMS	9.1350E-06	1.7000E-08	2
7964	IDMS	9.1220E-06	3.2000E-08	2
7965	IDMS	9.1300E-06	4.6000E-08	2
7967	IDMS	9.1790E-06	1.5000E-08	2
7969	IDMS	9.1740E-06	3.7000E-08	2
8130	IDMS	9.1667E-06	3.3000E-09	2
8131	IDMS	9.1783E-06	2.5000E-09	2
8132	IDMS	9.1600E-06	1.3200E-07	1

Laboratory	ITV: 0.18% z score	ITV: 0.28% z score	zeta score	ITV: 0.18% acceptable uncertainty for $\lVert zeta \rVert \leq 2$	ITV: 0.28% acceptable uncertainty for $\lVert zeta \rVert \leq 2$
7961	2.29	1.47	0.89	NO	YES
7962	-2.56	-1.65	-2.38	-	-
7964	-4.14	-2.66	-2.11	-	-
7965	-3.17	-2.04	-1.13	NO	YES
7967	2.78	1.79	2.90	-	-
7969	2.17	1.40	0.96	NO	YES
8130	1.29	0.83	3.54	-	-
8131	2.69	1.73	7.94	-	-
8132	0.47	0.30	0.03	NO	NO

REIMEP-17A: Plutonium and uranium amount content, and isotope amount ratios in synthetic input solution

Certified value for Pu amount content: 9.156 1 ± 0.005 0 µmol/g [$U=k \cdot u_c(k=2)$]

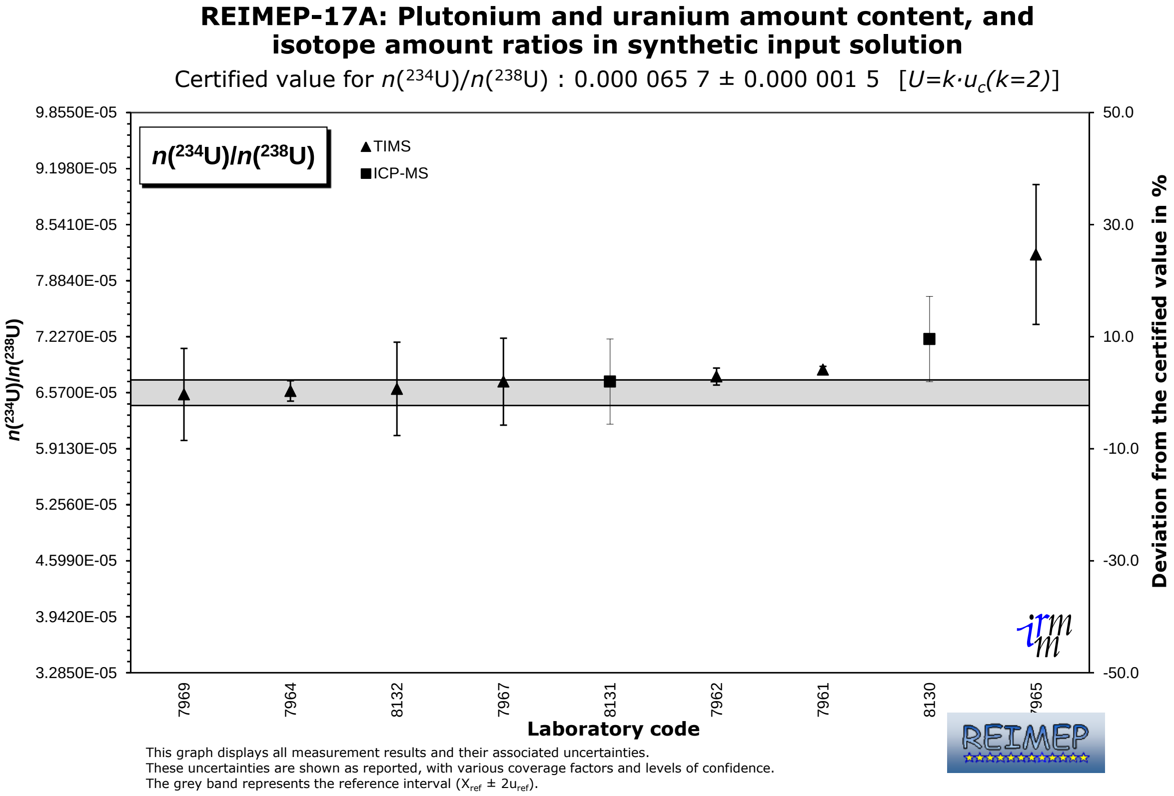


This graph displays all measurement results and their associated uncertainties.
These uncertainties are shown as reported, with various coverage factors and levels of confidence.
The grey band represents the reference interval ($X_{ref} \pm 2u_{ref}$).

Annex 12: Results for $n(^{234}U)/n(^{238}U)$ in REIMEP-17A

Laboratory	Analytical method	Reported $n(^{234}U)/n(^{238}U)$	Reported uncertainty $n(^{234}U)/n(^{238}U)$	Coverage factor *k*	zeta score
7961	TIMS	0.0000684	0.0000004	1	3.16
7962	TIMS	0.0000676	0.000001	2	2.10
7964	TIMS	0.0000659	0.0000012	2	0.21
7965	TIMS	0.0000819	0.0000082	2	3.89
7967	TIMS	0.000067	0.0000051	2	0.49
7969	TIMS	0.0000655	0.0000054	2	-0.07
8130	ICP-MS	0.000072	0.000005	2	2.41
8131	ICP-MS	0.000067	0.000005	2	0.50
8132	TIMS	0.00006615	0.00000547	2	0.16

As there are no ITVs defined for the minor uranium isotope ratios, there were no z scores and acceptable uncertainty scores issued for $n(^{234}U)/n(^{238}U)$.



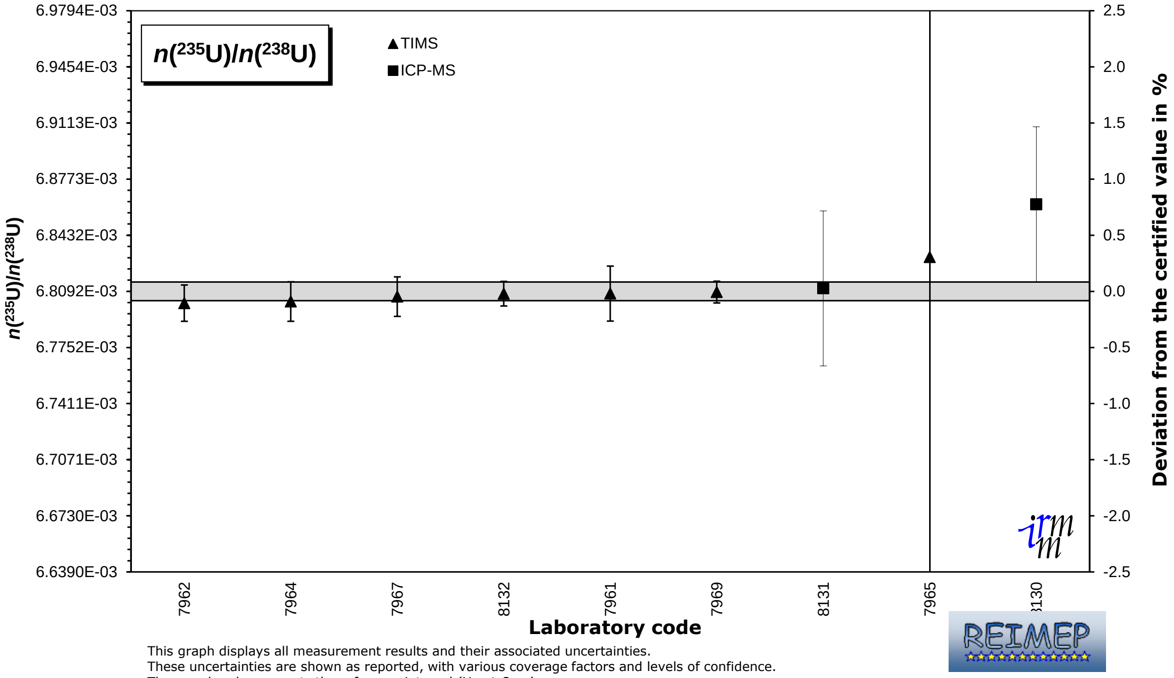
Annex 13: Results for $n(^{235}U)/n(^{238}U)$ in REIMEP-17A

Laboratory	Analytical method	Reported $n(^{235}U)/n(^{238}U)$	Reported uncertainty $n(^{235}U)/n(^{238}U)$	Coverage factor k
7961	TIMS	0.0068078	0.0000167	1
7962	TIMS	0.006802	0.000011	2
7964	TIMS	0.006803	0.000012	2
7965	TIMS	0.00683	0.00035	2
7967	TIMS	0.006806	0.000012	2
7969	TIMS	0.0068088	0.0000066	2
8130	ICP-MS	0.006862	0.000047	2
8131	ICP-MS	0.006811	0.000047	2
8132	TIMS	0.00680778	0.00000741	2

Laboratory	ITV: 0.28% z score	zeta score	ITV: 0.28% acceptable uncertainty for $\|zeta\| \leq 2$
7961	-0.15	-0.08	YES
7962	-0.76	-1.16	YES
7964	-0.65	-0.93	YES
7965	2.18	0.12	NO
7967	-0.34	-0.48	YES
7969	-0.04	-0.09	YES
8130	5.54	2.23	-
8131	0.19	0.08	NO
8132	-0.15	-0.30	YES

REIMEP-17A: Plutonium and uranium amount content, and isotope amount ratios in synthetic input solution

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

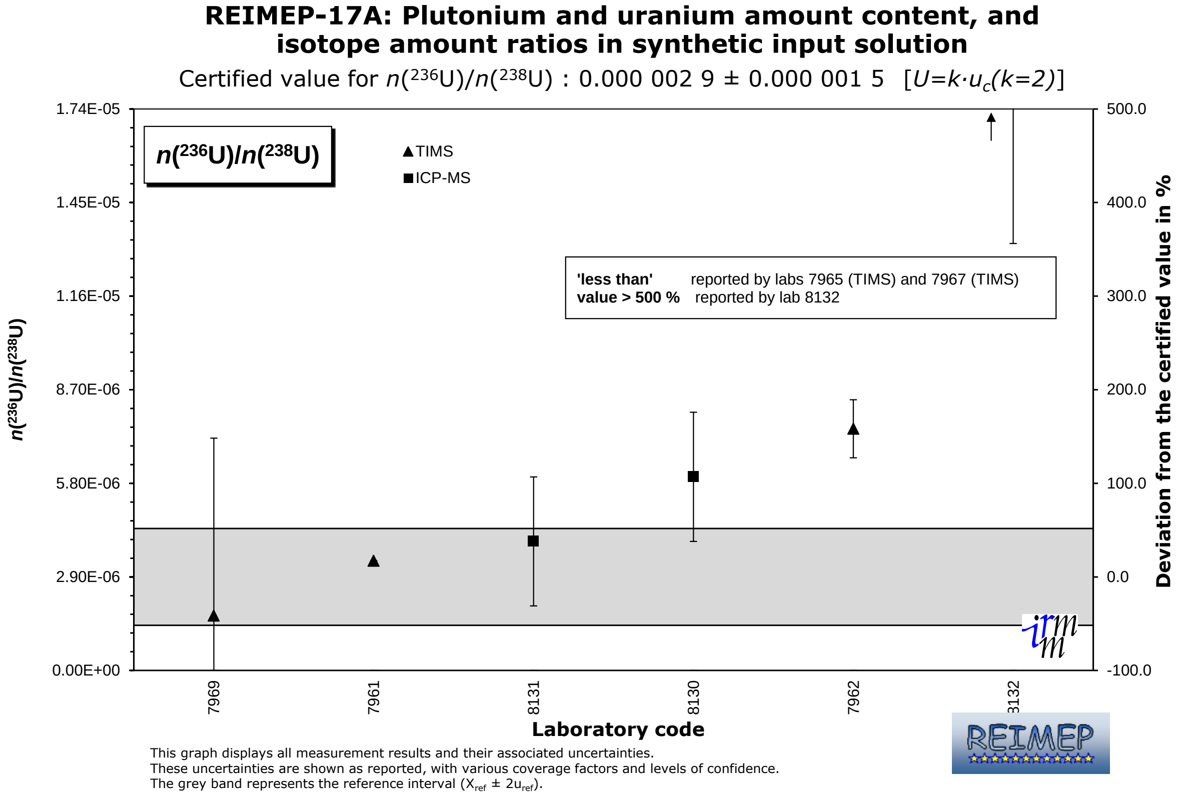


This graph displays all measurement results and their associated uncertainties.
These uncertainties are shown as reported, with various coverage factors and levels of confidence.
The grey band represents the reference interval ($X_{ref} \pm 2u_{ref}$).

Annex 14: Results for $n(^{236}U)/n(^{238}U)$ in REIMEP-17A

Laboratory	Analytical method	Reported $n(^{236}U)/n(^{238}U)$	Reported uncertainty $n(^{236}U)/n(^{238}U)$	Coverage factor *k*	zeta score
7961	TIMS	0.0000034	0	1	0.67
7962	TIMS	0.00000749	0.0000009	2	5.25
7964	-	-	-	-	-
7965	TIMS	<0.000079	-	-	-
7969	TIMS	0.0000017	0.0000055	2	-0.42
7967	TIMS	<0.00001	-	-	-
8130	ICP-MS	0.000006	0.000002	2	2.48
8131	ICP-MS	0.000004	0.000002	2	0.88
8132	TIMS	0.00001847	0.00000554	2	5.43

As there are no ITVs defined for the minor uranium isotope ratios, there were no z scores and acceptable uncertainty scores issued for $n(^{236}U)/n(^{238}U)$.



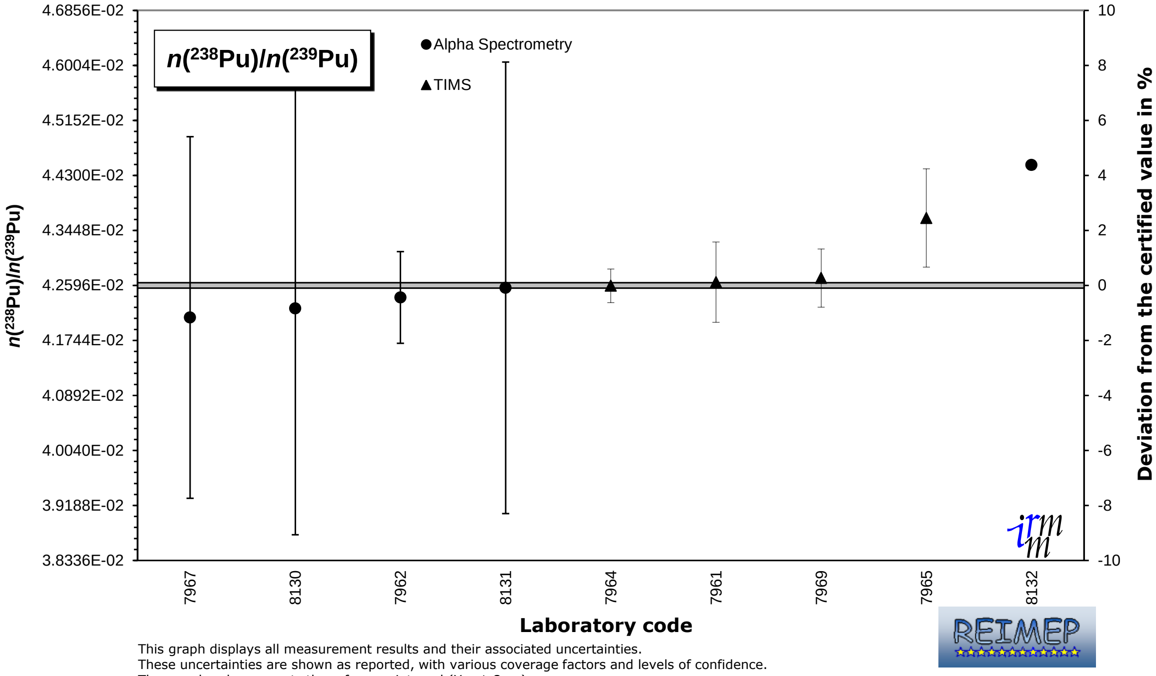
Annex 15: Results for $n(^{238}Pu)/n(^{239}Pu)$ in REIMEP-17A

Laboratory	Analytical method	Reported $n(^{238}Pu)/n(^{239}Pu)$	Reported uncertainty $n(^{238}Pu)/n(^{239}Pu)$	Coverage factor k
7961	TIMS	0.0426466	0.0006217	1
7962	Alpha spectrometry	0.04241	0.00071	2
7964	TIMS	0.04259	0.00026	2
7965	TIMS	0.04364	0.00076	2
7967	Alpha spectrometry	0.0421	0.0028	2
7969	TIMS	0.04271	0.00045	2
8130	Alpha spectrometry	0.042241	0.003506	2
8131	Alpha spectrometry	0.042559	0.003497	2
8132	Alpha spectrometry	0.04446	0.0000654	2

Laboratory	ITV: 1.8% z score	zeta score	ITV: 1.8% acceptable uncertainty for $\lVert zeta \rVert \leq 2$
7961	0.13	0.08	YES
7962	-0.49	-0.52	YES
7964	-0.02	-0.05	YES
7965	2.72	2.74	-
7967	-1.29	-0.35	NO
7969	0.30	0.50	YES
8130	-0.93	-0.20	NO
8131	-0.10	-0.02	NO
8132	4.86	47.96	-

REIMEP-17A: Plutonium and uranium amount content, and isotope amount ratios in synthetic input solution

Certified value for $n(^{238}Pu)/n(^{239}Pu)$: 0.042 596 ± 0.000 042 [$U=k \cdot u_c(k=2)$]



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} ± $2u_{ref}$).

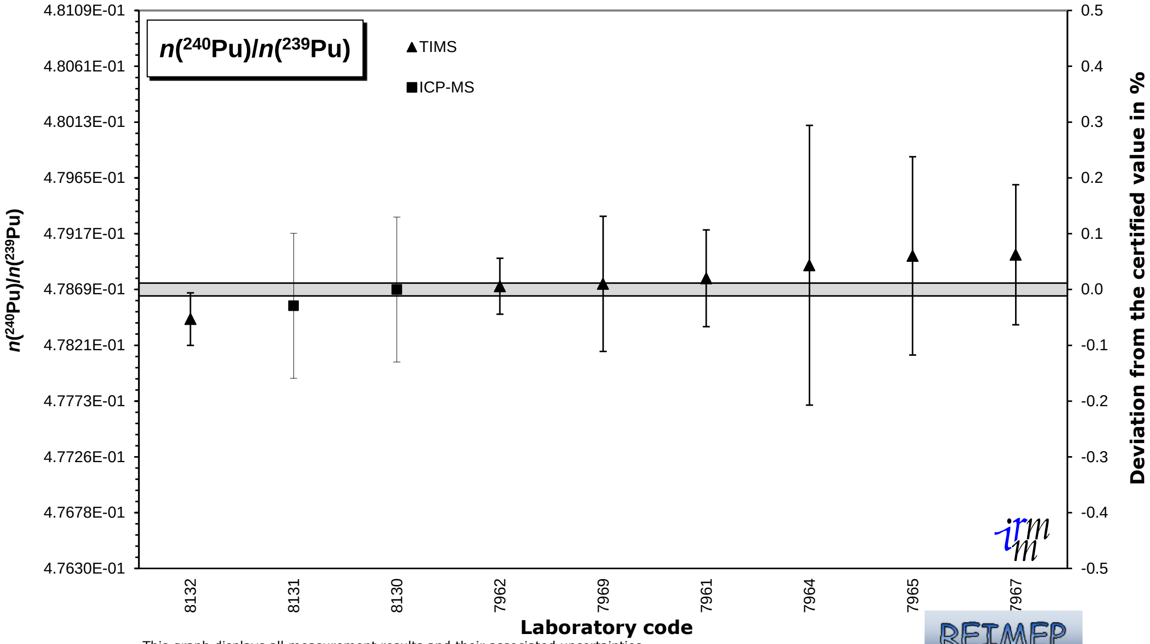
Annex 16: Results for $n(^{240}Pu)/n(^{239}Pu)$ in REIMEP-17A

Laboratory	Analytical method	Reported $n(^{240}Pu)/n(^{239}Pu)$	Reported uncertainty $n(^{240}Pu)/n(^{239}Pu)$	Coverage factor k
7961	TIMS	0.4787879	0.0004146	1
7962	TIMS	0.47872	0.00024	2
7964	TIMS	0.4789	0.0012	2
7965	TIMS	0.47898	0.00085	2
7967	TIMS	0.47899	0.0006	2
7969	TIMS	0.47874	0.00058	2
8130	ICP-MS	0.478692	0.000622	2
8131	ICP-MS	0.478552	0.000622	2
8132	TIMS	0.4784381	0.000225	2

Laboratory	ITV: 0.11% z score	zeta score	ITV: 0.11% acceptable uncertainty for $\|zeta\| \leq 2$
7961	0.36	0.23	YES
7962	0.11	0.23	YES
7964	0.79	0.35	NO
7965	1.09	0.68	YES
7967	1.13	0.99	YES
7969	0.18	0.16	YES
8130	0.00	0.00	YES
8131	-0.53	-0.45	YES
8132	-0.96	-2.19	-

REIMEP-17A: Plutonium and uranium amount content, and isotope amount ratios in synthetic input 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} ± $2u_{ref}$).

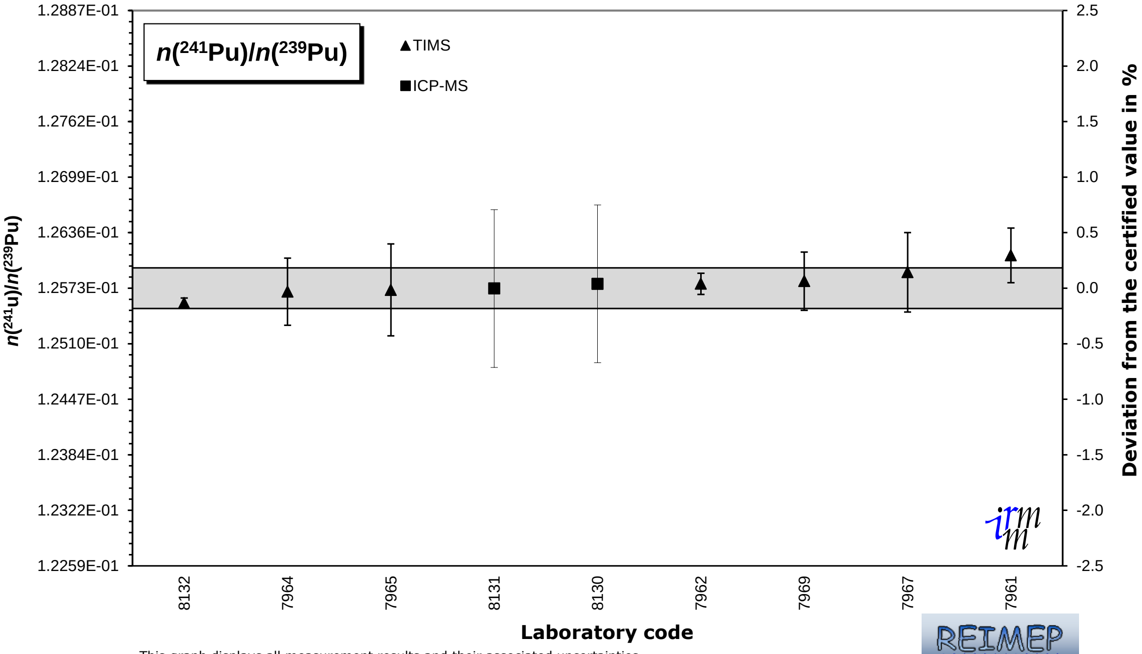
Annex 17: Results for $n(^{241}Pu)/n(^{239}Pu)$ in REIMEP-17A

Laboratory	Analytical method	Reported $n(^{241}Pu)/n(^{239}Pu)$	Reported uncertainty $n(^{241}Pu)/n(^{239}Pu)$	Coverage factor k
7961	TIMS	0.1261019	0.0003089	1
7962	TIMS	0.12578	0.00012	2
7964	TIMS	0.12569	0.00038	2
7965	TIMS	0.12571	0.00052	2
7967	TIMS	0.12591	0.00045	2
7969	TIMS	0.12581	0.00033	2
8130	ICP-MS	0.125779	0.000893	2
8131	ICP-MS	0.125726	0.000893	2
8132	TIMS	0.125561	0.000058	2

Laboratory	ITV: 0.28% z score	zeta score	ITV: 0.28% acceptable uncertainty for $\|\text{zeta}\| \leq 2$
7961	2.11	1.13	YES
7962	0.28	0.39	YES
7964	-0.23	-0.18	YES
7965	-0.11	-0.07	YES
7967	1.02	0.71	YES
7969	0.45	0.40	YES
8130	0.28	0.11	NO
8131	-0.02	-0.01	NO
8132	-0.96	-1.42	YES

REIMEP-17A: Plutonium and uranium amount content, and isotope amount ratios in sythetic input 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 ($X_{ref} \pm 2u_{ref}$).

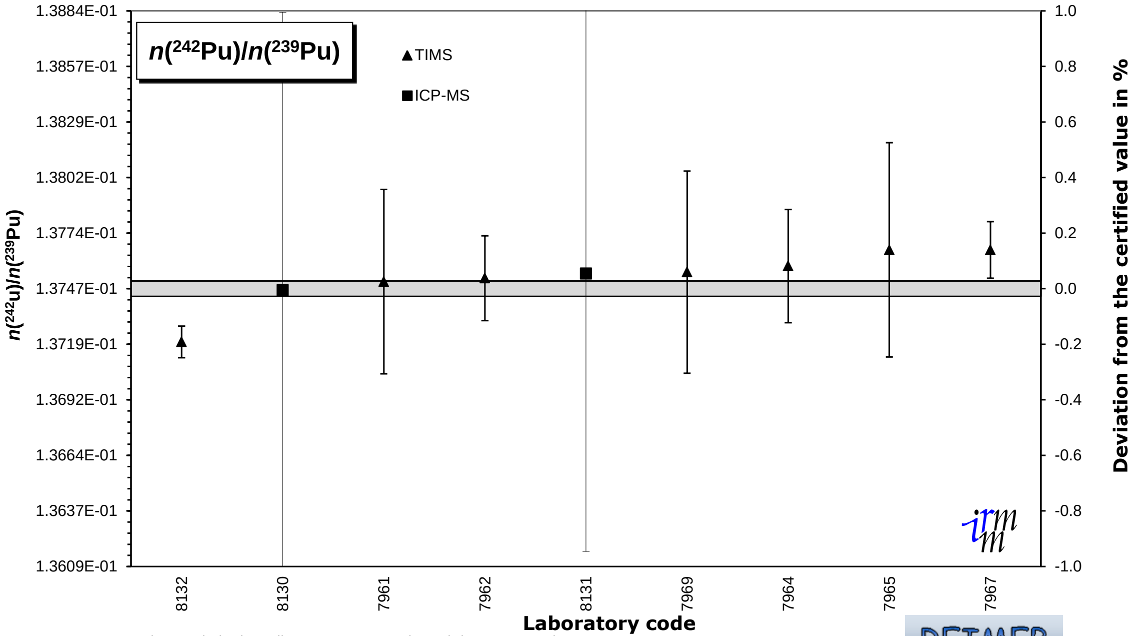
Annex 18: Results for $n(^{242}Pu)/n(^{239}Pu)$ in REIMEP-17A

Laboratory	Analytical method	Reported $n(^{242}Pu)/n(^{239}Pu)$	Reported uncertainty $n(^{242}Pu)/n(^{239}Pu)$	Coverage factor k
7961	TIMS	0.1375029	0.000456	1
7962	TIMS	0.13752	0.00021	2
7964	TIMS	0.13758	0.00028	2
7965	TIMS	0.13766	0.00053	2
7967	TIMS	0.13766	0.00014	2
7969	TIMS	0.13755	0.0005	2
8130	ICP-MS	0.13746	0.001375	2
8131	ICP-MS	0.137543	0.001375	2
8132	TIMS	0.1372046	0.0000783	2

Laboratory	ITV: 0.36% z score	zeta score	ITV: 0.36% acceptable uncertainty for $\lVert \text{zeta} \rVert \leq 2$
7961	0.14	0.08	YES
7962	0.21	0.49	YES
7964	0.45	0.79	YES
7965	0.78	0.72	YES
7967	0.78	2.65	-
7969	0.33	0.33	YES
8130	-0.03	-0.01	NO
8131	0.30	0.11	NO
8132	-1.06	-6.05	-

REIMEP-17A: Plutonium and uranium amount content, and isotope amount ratios in sythetic input 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 ($X_{ref} \pm 2u_{ref}$).

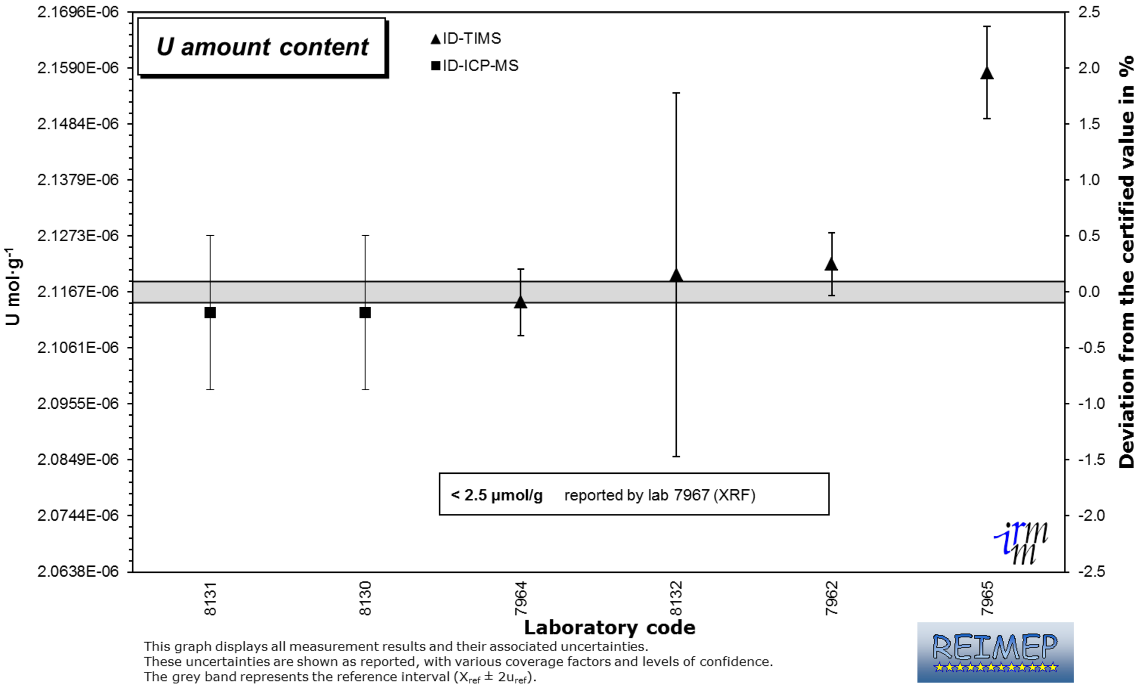
Annex 19: Results for the uranium amount content in REIMEP-17B

Laboratory	Analytical method	Reported uranium content $mol \cdot g^{-1}$	Reported uncertainty uranium content $mol \cdot g^{-1}$	Coverage factor *k*
7961	-	-	-	-
7962	IDMS	2.1220E-06	5.9000E-09	2
7964	IDMS	2.1148E-06	6.3000E-09	2
7965	IDMS	2.1582E-06	8.7000E-09	2
7967	XRF	<2.5E-06	-	-
7969	-	-	-	-
8130	IDMS	2.1128E-06	1.4580E-08	2
8131	IDMS	2.1128E-06	1.4580E-08	2
8132	IDMS	2.12E-06	3.44E-08	1

Laboratory	ITV: 0.18% z score	ITV: 0.28% z score	zeta score	ITV: 0.18% acceptable uncertainty for $\|zeta\| \leq 2$	ITV: 0.28% acceptable uncertainty for $\|zeta\| \leq 2$
7961	-	-	-	-	-
7962	2.78	1.79	1.70	YES	YES
7964	-1.00	-0.64	-0.57	YES	YES
7965	21.78	14.00	9.30	-	-
7967	-	-	-	-	-
7969	-	-	-	-	-
8130	-2.04	-1.31	-0.53	NO	NO
8131	-2.05	-1.32	-0.53	NO	NO
8132	1.73	1.11	0.10	NO	NO

REIMEP-17B: Plutonium and uranium amount content, and isotope amount ratios in synthetic input solution

Certified value for U amount content: 2.116 7 ± 0.002 0 µmol/g [$U=k\cdot u_c(k=2)$]



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 ($X_{ref} \pm 2u_{ref}$).

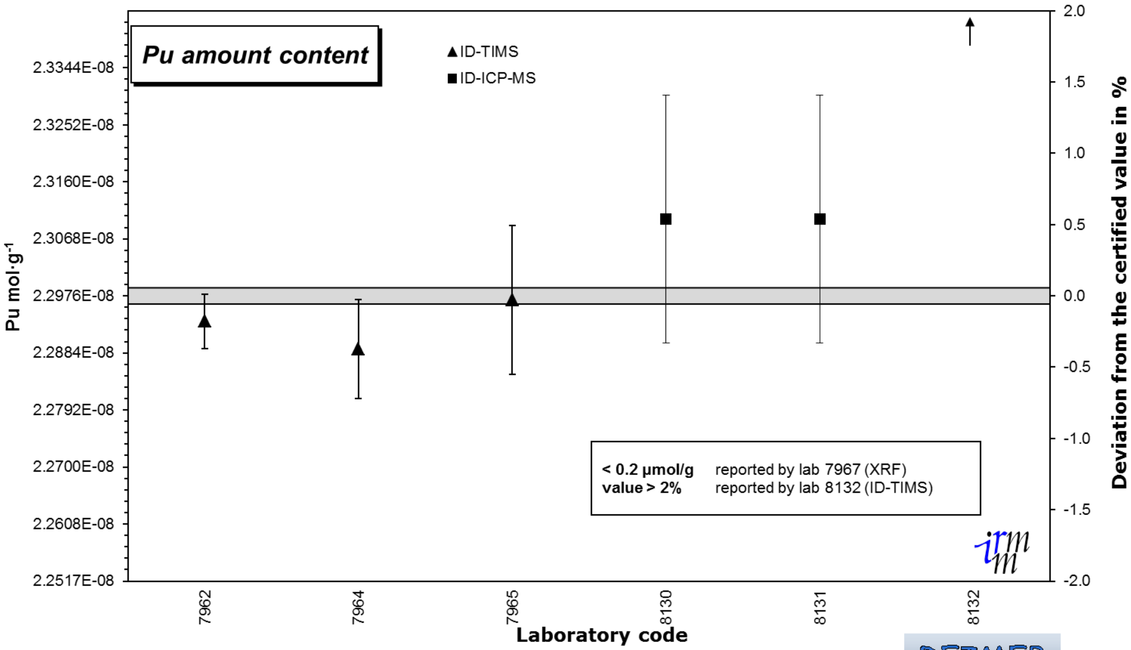
Annex 20: Results for the plutonium amount content in REIMEP-17B

Laboratory	Analytical method	Reported plutonium content *mol·g*$^{-1}$	Reported uncertainty plutonium content *mol·g*$^{-1}$	Coverage factor *k*
7961	-	-	-	-
7962	IDMS	2.2935E-08	4.4000E-11	2
7964	IDMS	2.2890E-08	8.0000E-11	2
7965	IDMS	2.2970E-08	1.2000E-10	2
7967	XRF	<0.2E-06	-	-
7969	-	-	-	-
8130	IDMS	2.3100E-08	2.0000E-10	2
8131	IDMS	2.3100E-08	2.0000E-10	2
8132	IDMS	3.2850E-06	4.7300E-08	1

Laboratory	ITV: 0.18% z score	ITV: 0.28% z score	zeta score	ITV: 0.18% acceptable uncertainty for $\|zeta\| \leq 2$	ITV: 0.28% acceptable uncertainty for $\|zeta\| \leq 2$
7961	-	-	-	-	-
7962	-1.98	-1.27	-1.79	YES	YES
7964	-4.16	-2.67	-2.12	-	-
7965	-0.29	-0.19	-0.10	NO	YES
7967	-	-	-	-	-
7969	-	-	-	-	-
8130	6.00	3.85	1.24	NO	NO
8131	6.00	3.85	1.24	NO	NO
8132	157750.31	101410.91	68.96	-	-

REIMEP-17B: Plutonium and uranium amount content, and isotope amount ratios in synthetic input solution

Certified value for Pu amount content: 0.022 976 ± 0.000 013 µmol/g [$U=k\cdot u_c(k=2)$]

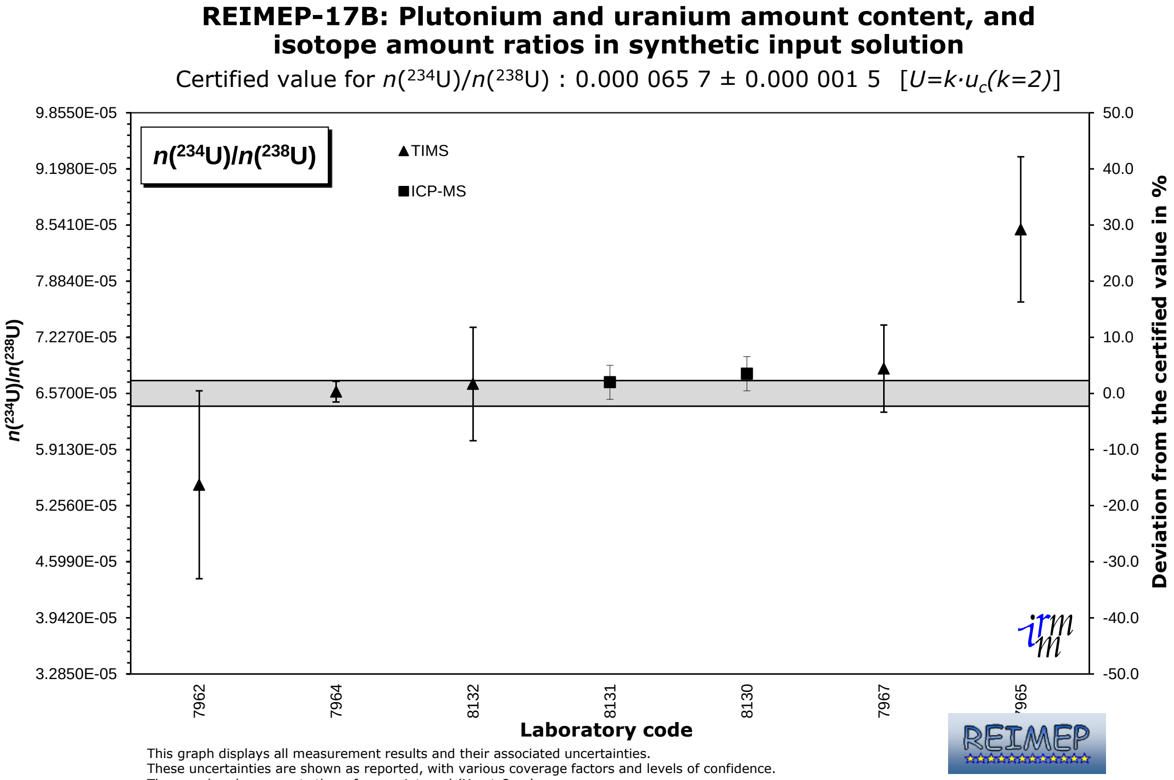


This graph displays all measurement results and their associated uncertainties.
These uncertainties are shown as reported, with various coverage factors and levels of confidence.
The grey band represents the reference interval ($X_{ref} \pm 2u_{ref}$).

Annex 21: Results for $n(^{234}U)/n(^{238}U)$ in REIMEP-17B

Laboratory	Analytical method	Reported $n(^{234}U)/n(^{238}U)$	Reported uncertainty $n(^{234}U)/n(^{238}U)$	Coverage factor *k*	zeta score
7961	-	-	-	-	-
7962	TIMS	0.000055	0.000011	2	-1.93
7964	TIMS	0.0000659	0.0000012	2	0.21
7965	TIMS	0.0000849	0.0000085	2	4.45
7967	TIMS	0.0000686	0.0000051	2	1.09
7969	-	-	-	-	-
8130	ICP-MS	0.000068	0.000002	2	1.84
8131	ICP-MS	0.000067	0.000002	2	1.04
8132	TIMS	0.0000668	0.00000663	2	0.32

As there are no ITVs defined for the minor uranium isotope ratios, there were no z scores and acceptable uncertainty scores issued for $n(^{234}U)/n(^{238}U)$.



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 ($X_{ref} \pm 2u_{ref}$).

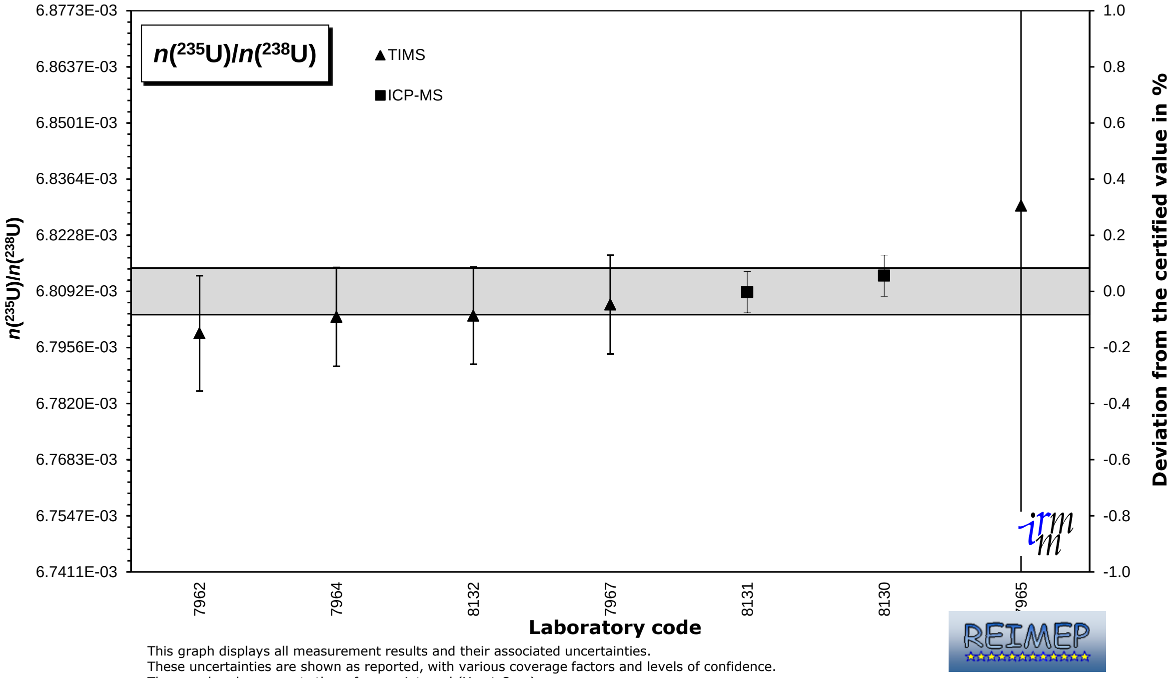
Annex 22: Results for $n(^{235}U)/n(^{238}U)$ in REIMEP-17B

Laboratory	Analytical method	Reported $n(^{235}U)/n(^{238}U)$	Reported uncertainty $n(^{235}U)/n(^{238}U)$	Coverage factor k
7961	-	-	-	-
7962	TIMS	0.006799	0.000014	2
7964	TIMS	0.006803	0.000012	2
7965	TIMS	0.00683	0.00036	2
7967	TIMS	0.006806	0.000012	2
7969	-	-	-	-
8130	ICP-MS	0.006813	0.000005	2
8131	ICP-MS	0.006809	0.000005	2
8132	TIMS	0.0068033	0.0000118	2

Laboratory	ITV: 0.28% z score	zeta score	ITV: 0.28% acceptable uncertainty for $\|zeta\| \leq 2$
7961	-	-	-
7962	-1.07	-1.35	YES
7964	-0.65	-0.93	YES
7965	2.18	0.12	NO
7967	-0.34	-0.48	YES
7969	-	-	-
8130	0.40	1.00	YES
8131	-0.02	-0.05	YES
8132	-0.62	-0.90	YES

REIMEP-17B: Plutonium and uranium amount content, and isotope amount ratios in synthetic input solution

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

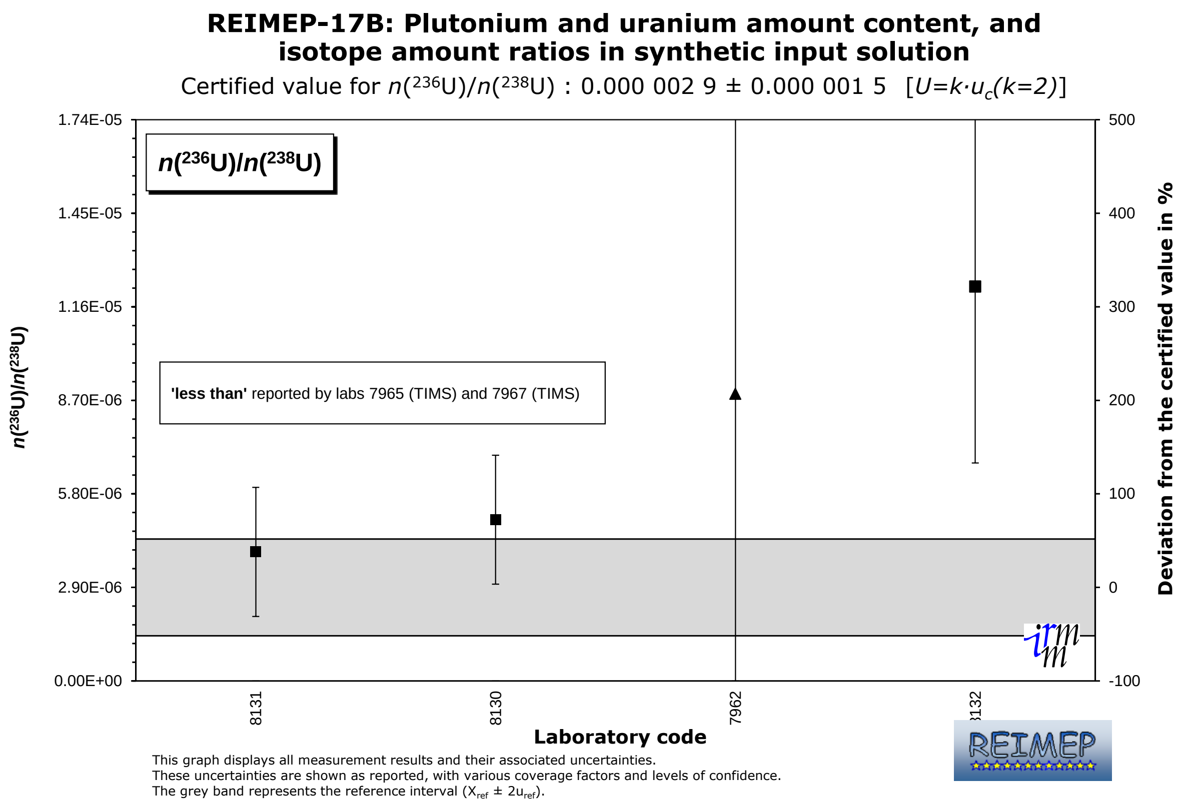


This graph displays all measurement results and their associated uncertainties.
These uncertainties are shown as reported, with various coverage factors and levels of confidence.
The grey band represents the reference interval ($X_{ref} \pm 2u_{ref}$).

Annex 23: Results for $n(^{236}U)/n(^{238}U)$ in REIMEP-17B

Laboratory	Analytical method	Reported $n(^{236}U)/n(^{238}U)$	Reported uncertainty $n(^{236}U)/n(^{238}U)$	Coverage factor k	zeta score
7961	-	-	-	-	-
7962	TIMS	0.0000089	0.000012	2	0.99
7964	-	-	-	-	-
7965	TIMS	<0.000084	-	-	-
7967	TIMS	<0.00001	-	-	-
7969	-	-	-	-	-
8130	ICP-MS	0.000005	0.000002	2	1.68
8131	ICP-MS	0.000004	0.000002	2	0.88
8132	TIMS	0.00001223	0.00000547	2	3.29

As there are no ITVs defined for the minor uranium isotope ratios, there were no z scores and acceptable uncertainty scores issued for $n(^{236}U)/n(^{238}U)$.



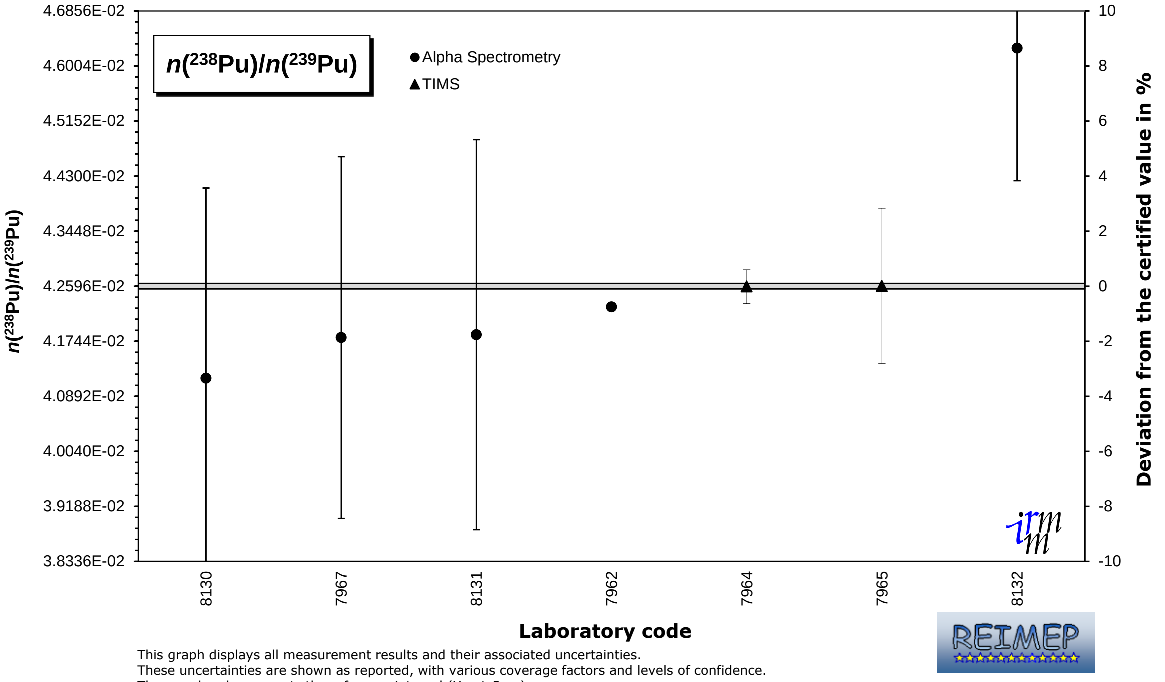
Annex 24: Results for $n(^{238}Pu)/n(^{239}Pu)$ in REIMEP-17B

Laboratory	Analytical method	Reported $n(^{238}Pu)/n(^{239}Pu)$	Reported uncertainty $n(^{238}Pu)/n(^{239}Pu)$	Coverage factor k
7961	-	-	-	-
7962	Alpha spectrometry	0.042276	0.000034	2
7964	TIMS	0.04259	0.00026	2
7965	TIMS	0.0426	0.0012	2
7967	Alpha spectrometry	0.0418	0.0028	2
7969	-	-	-	-
8130	Alpha spectrometry	0.041171	0.002943	2
8131	Alpha spectrometry	0.041845	0.003018	2
8132	Alpha spectrometry	0.04628	0.00205	2

Laboratory	ITV: 1.8% z score	zeta score	ITV: 1.8% acceptable uncertainty for $\|zeta\| \leq 2$
7961	-	-	-
7962	-0.83	-11.84	-
7964	-0.02	-0.05	YES
7965	0.01	0.01	YES
7967	-2.08	-0.57	NO
7969	-	-	-
8130	-3.72	-0.97	NO
8131	-1.96	-0.50	NO
8132	9.61	3.59	-

REIMEP-17B: Plutonium and uranium amount content, and isotope amount ratios in synthetic input solution

Certified value for $n(^{238}Pu)/n(^{239}Pu)$: 0.042 596 ± 0.000 042 [$U=k\cdot u_c(k=2)$]



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} ± $2u_{ref}$).

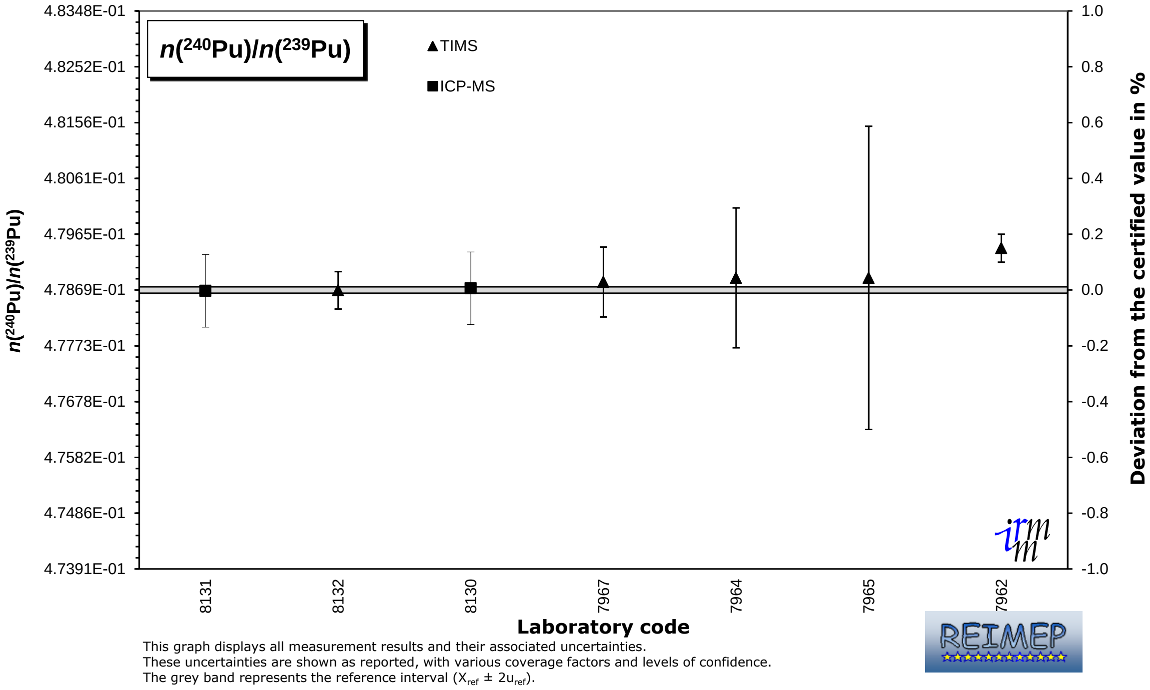
Annex 25: Results for $n(^{240}Pu)/n(^{239}Pu)$ in REIMEP-17B

Laboratory	Analytical method	Reported $n(^{240}Pu)/n(^{239}Pu)$	Reported uncertainty $n(^{240}Pu)/n(^{239}Pu)$	Coverage factor k
7961	-	-	-	-
7962	TIMS	0.47941	0.00024	2
7964	TIMS	0.4789	0.0012	2
7965	TIMS	0.4789	0.0026	2
7967	TIMS	0.47883	0.0006	2
7969	-	-	-	-
8130	ICP-MS	0.478722	0.000622	2
8131	ICP-MS	0.478677	0.000622	2
8132	TIMS	0.478687	0.000321	2

Laboratory	ITV: 0.11% z score	zeta score	ITV: 0.11% acceptable uncertainty for $\|zeta\| \leq 2$
7961	-	-	-
7962	2.73	5.83	-
7964	0.79	0.35	NO
7965	0.79	0.16	NO
7967	0.52	0.46	YES
7969	-	-	-
8130	0.11	0.10	YES
8131	-0.06	-0.05	YES
8132	-0.02	-0.03	YES

REIMEP-17B: Plutonium and uranium amount content, and isotope amount ratios in synthetic input 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 ($X_{ref} \pm 2u_{ref}$).

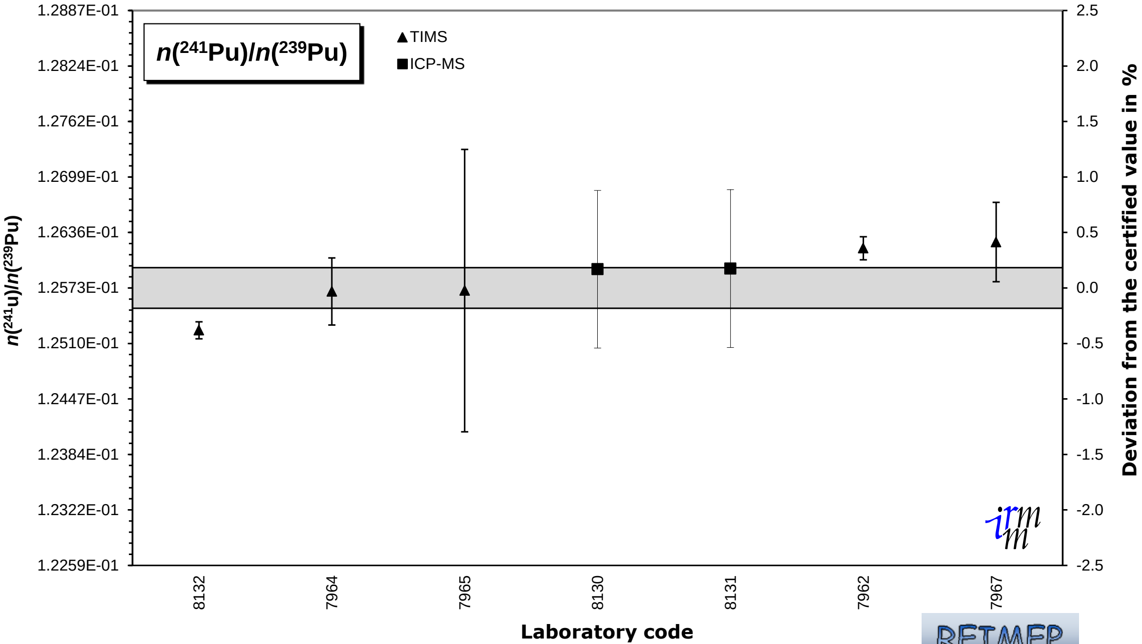
Annex 26: Results for $n(^{241}Pu)/n(^{239}Pu)$ in REIMEP-17B

Laboratory	Analytical method	Reported $n(^{241}Pu)/n(^{239}Pu)$	Reported uncertainty $n(^{241}Pu)/n(^{239}Pu)$	Coverage factor k
7961	-	-	-	-
7962	TIMS	0.12618	0.00013	2
7964	TIMS	0.12569	0.00038	2
7965	TIMS	0.1257	0.0016	2
7967	TIMS	0.12625	0.00045	2
7969	-	-	-	-
8130	ICP-MS	0.125942	0.000894	2
8131	ICP-MS	0.12595	0.000894	2
8132	TIMS	0.1252507	0.0000962	2

Laboratory	ITV: 0.28% z score	zeta score	ITV: 0.28% acceptable uncertainty for $\lVert zeta \rVert \leq 2$
7961	-	-	-
7962	2.56	3.41	-
7964	-0.23	-0.18	YES
7965	-0.17	-0.04	NO
7967	2.95	2.06	-
7969	-	-	-
8130	1.20	0.46	NO
8131	1.25	0.48	NO
8132	-2.72	-3.85	-

REIMEP-17B: Plutonium and uranium amount content, and isotope amount ratios in sythetic input 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 ($X_{ref} \pm 2u_{ref}$).

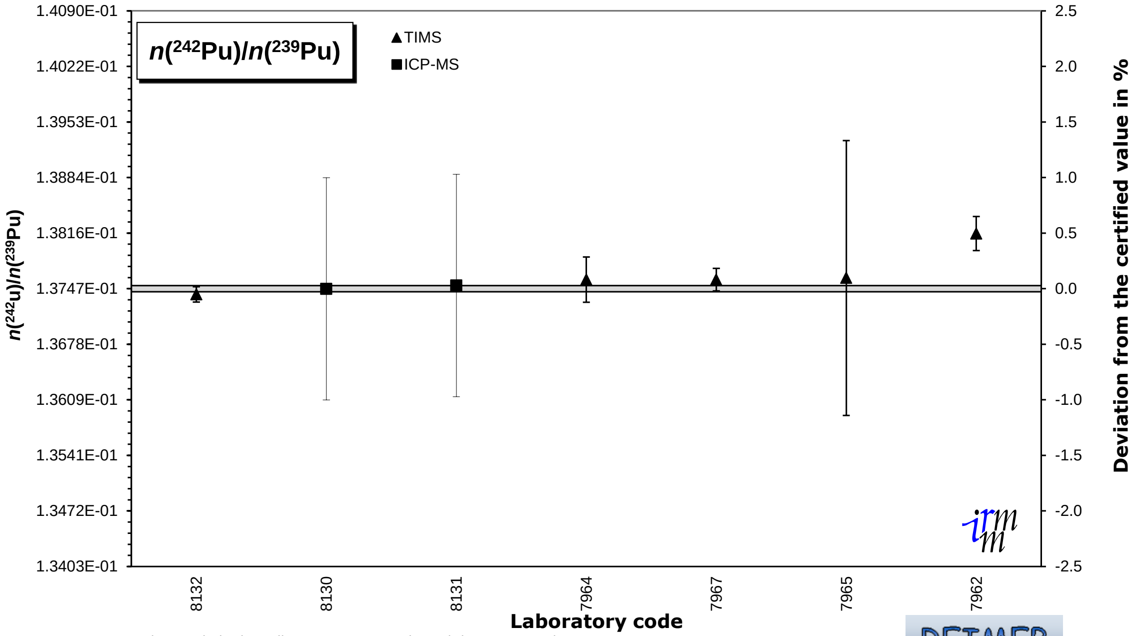
Annex 27: Results for $n(^{242}Pu)/n(^{239}Pu)$ in REIMEP-17B

Laboratory	Analytical method	Reported $n(^{242}Pu)/n(^{239}Pu)$	Reported uncertainty $n(^{242}Pu)/n(^{239}Pu)$	Coverage factor k
7961	-	-	-	-
7962	TIMS	0.13815	0.00021	2
7964	TIMS	0.13758	0.00028	2
7965	TIMS	0.1376	0.0017	2
7967	TIMS	0.13758	0.00014	2
7969	-	-	-	-
8130	ICP-MS	0.137466	0.001375	2
8131	ICP-MS	0.137507	0.001375	2
8132	TIMS	0.1373978	0.0000944	2

Laboratory	ITV: 0.36% z score	zeta score	ITV: 0.36% acceptable uncertainty for $\lVert zeta \rVert \leq 2$
7961	-	-	-
7962	2.76	6.39	-
7964	0.45	0.79	YES
7965	0.53	0.16	NO
7967	0.45	1.54	YES
7969	-	-	-
8130	-0.01	0.00	NO
8131	0.16	0.06	NO
8132	-0.28	-1.38	YES

REIMEP-17B: Plutonium and uranium amount content, and isotope amount ratios in sythetic input 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 ($X_{ref} \pm 2u_{ref}$).

Annex 28: Summary of the information given by the participants from the questionnaire

Laboratory	How did you determine the U, Pu content in REIMEP-17 samples?
7961	IDMS
7962	IDMS
7964	IDMS
7965	IDMS
7967	IDMS, K-edge, XRF
7969	IDMS
8130	IDMS
8131	IDMS
8132	IDMS

Laboratory	Did you perform a chemical separation prior to measurement?	Which resin?
7961	YES	TOPO
7962	YES	AG1X4
7964	YES	AG1X4
7965	YES	TEVA
7967	YES	UTEVA
7969	YES	TOPO
8130	YES	UTEVA
8131	YES	TEVA
8132	YES	AG1X2

Laboratory	Did you use alpha spectrometry to measure isotope ratios?	Which source preparation technique did you apply?
7961	NO	
7962	YES	drop deposition
7964	NO	
7965	NO	
7967	YES	drop deposition
7969	NO	
8130	YES	drop deposition
8131	YES	drop deposition
8132	YES	drop deposition

Laboratory	Did you use a mass-spectrometric technique to measure isotope ratios?	Did you apply a correction for mass fractionation?
7961	TIMS	NO
7962	TIMS	YES, using standards
7964	TIMS	YES, total evaporation and standards
7965	TIMS	NO
7967	TIMS	NO
7969	TIMS	YES, standards (bracketing) and total evaporation
8130	ICP-MS	YES, standards
8131	ICP-MS	YES, standards
8132	TIMS	YES, standards

Laboratory	Describe the mass spectrometer used?	Detector
7961	Triton	Faraday cups
7962	VG sector 54	Faraday cups
7964	VG sector 54, VG 354	Faraday cups, Daly detector
7965	Triton	Faraday cups
7967	MAT 261	Faraday cups
7969	Triton	Faraday cups
8130	NU Instrument, VG Sector 54-30	Faraday cups
8131	NU Instrument, VG Sector 54-30	Faraday cups
8132	VG Isomass 54E	Faraday cups

Laboratory	How did you estimate measurement uncertainty?
7961	Derived from ITV-2010
7962	GUM, bottom up approach
7964	GUM
7965	Estimated on analysis of standards
7967	GUM
7969	GUM
8130	Duplicate pairs and method QCS
8131	Duplicate and method QCS
8132	GUM

Laboratory	Does your laboratory use CRMs?	CRMs and suppliers
7961	YES	NBL010, NBL030, NBL128,
7962	YES	NBL137 NBS 947, IRMM-040a, IRMM-054, IRMM-083, IRMM-046b, IRMM-049c, NBS 950, NBS 005 through NBS 900 series
7964	YES	CETAMA, NBS series (U010, U500), IRMM series (184, 186, 199)
7965	YES	CETAMA, IRMM, NBL
7967	YES	IRMM-185, IRMM-290, LSD 1027
7969	YES	NBL, IRMM, KRI, CETAMA
8130	YES	CRM137, NBS010, NBS020, NBS200, NBL
8131	YES	CRM137, NBS010, NBS020, NBS200, NBL
8132	YES	NBS500, NBS005, NBS020, NBS050, NBS350, NBS750, NBS930, NBS960, NBS947

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