

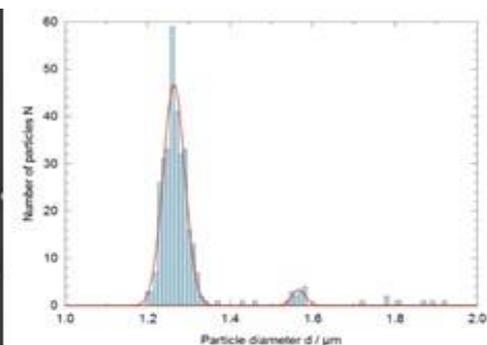
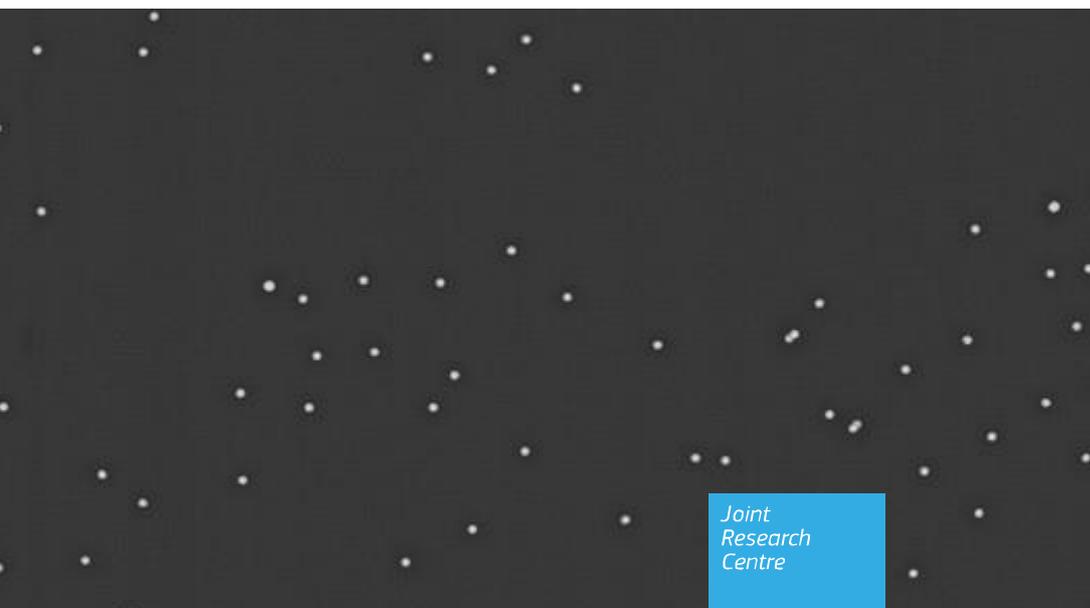
JRC REFERENCE MATERIALS REPORT

Preparation and Certification of the Uranium Oxide Micro Particles IRMM-2329P

*Certified for Isotope Ratios
and Uranium Amount per
Particle*

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2020



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Foreword

The Directorate G "Nuclear Safety and Security", Unit G.2 "Standards for Nuclear Safety, Security and Safeguards" (SN3S) at the European Commission's Joint Research Centre in Geel, Belgium (formerly known as the "Institute for Reference Materials and Measurements" IRMM), provides a wide range of nuclear Certified Reference Materials (CRMs) to safeguards authorities and the nuclear industry.

This report describes the certification of the IRMM-2329P, a uranium micro particle reference material. The project was a collaboration between the Forschungszentrum Jülich (FZJ) GmbH, Germany, and the International Atomic Energy Agency (IAEA), Austria.

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Abstract

This report describes the certification of the IRMM-2329P, a uranium micro particle reference material.

This certification project was a collaboration between the International Atomic Energy Agency (IAEA-SGAS, Austria), the Forschungszentrum Jülich GmbH (FZJ, Germany) and the Joint Research Centre (JRC-Geel, Unit G.2).

The material was produced in compliance with ISO/IEC 17034:2016 [1] and certified in accordance with ISO Guide 35:2006 [2]. Between-unit homogeneity was quantified and stability during dispatch and storage were investigated in accordance with ISO Guide 35:2006 [2]. Uncertainties of the certified values were estimated in compliance with ISO/IEC Guide 98-3:2008 (the Guide to the Expression of Uncertainty in Measurement, GUM) [3]. In order to achieve "fit for purpose" isotope ratios for particle analysis, the base material IRMM-2329 was prepared by mixing of two uranium nitrate solutions, which were prepared by hydrolysis and nitration of uranium hexafluoride (UF₆) CRM's. Certification of the uranium isotopic composition was done by Thermal Ionisation Mass Spectrometry (TIMS) using the Modified Total Evaporation (MTE) method.

Production of the uranium particles was done at the FZJ using a dedicated method based on spray-pyrolysis of droplets generated with a vibrating orifice aerosol generator (VOAG). Verification measurements of the base solution and 'process control measurements' on the uranium particles were performed at the IAEA-SGAS via Multi Collector Inductively Coupled Plasma Mass Spectrometry (MC-ICPMS). In addition to the isotopic composition, the uranium amount and mass per particle is certified by Isotope Dilution Thermal Ionisation Mass Spectrometry (ID-TIMS) and confirmed by ID-MC-ICPMS performed at IAEA-SGAS.

Moreover, the certified values were established using new published values for the atomic masses and applying a 2-digit rounding rule.

The materials are intended for the calibration of instruments and methods, quality control purposes, and the assessment of method performance for isotope mass spectrometry on uranium particles. As with any certified reference material, they can also be used for validation studies.

The following values were assigned to the IRMM-2329P:

TRIURANIUM OCTOXIDE PARTICLE		
	Isotope amount ratio	
	Certified value ¹⁾ [mol/mol]	Uncertainty ²⁾ [mol/mol]
$n(^{234}\text{U})/n(^{238}\text{U})$	0.00034083	0.00000019
$n(^{235}\text{U})/n(^{238}\text{U})$	0.033902	0.000012
$n(^{236}\text{U})/n(^{238}\text{U})$	0.00003021	0.00000012
	Isotope amount fraction	
	Certified value ³⁾ [mol/mol]	Uncertainty ²⁾ [mol/mol]
$n(^{234}\text{U})/n(\text{U})$	0.00032953	0.00000018
$n(^{235}\text{U})/n(\text{U})$	0.032778	0.000011
$n(^{236}\text{U})/n(\text{U})$	0.00002921	0.00000012
$n(^{238}\text{U})/n(\text{U})$	0.966863	0.000011
	Isotope mass fraction	
	Certified value ^{3) 4)} [g/g]	Uncertainty ²⁾ [g/g]
$m(^{234}\text{U})/m(\text{U})$	0.00032412	0.00000018
$m(^{235}\text{U})/m(\text{U})$	0.032378	0.000011

$m(^{236}\text{U})/m(\text{U})$	0.00002897	0.00000012
$m(^{238}\text{U})/m(\text{U})$	0.967269	0.000011
	Molar mass	
	Certified value ^{3) 4)} [g/mol]	Uncertainty ²⁾ [g/mol]
$M(\text{U})$	237.950848	0.000034
	Uranium amount per particle	
	Certified value [fmol]	Uncertainty ²⁾ [fmol]
$n(\text{U})$	15.1	2.6
	Uranium mass per particle	
	Certified value [pg]	Uncertainty ²⁾ [pg]
$m(\text{U})$	3.58	0.63
	Number of uranium atoms per particle	
	Certified value	Uncertainty ²⁾
$N(\text{U})$	$9.1 \cdot 10^9$	$1.6 \cdot 10^9$

1) The certified values are traceable to the International System of units (SI) via IRMM-074.

2) The 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.

3) These values are calculated using the isotope amount ratios and therefore traceable to the SI. The calculation of $n(\text{U})$, $m(\text{U})$ and $M(\text{U})$ includes the contributions from the isotopes ^{234}U , ^{235}U , ^{236}U and ^{238}U .

4) These values are calculated using the values listed below from Wang *et al.*, *The AME 2016 atomic mass evaluation, Chinese Physics C Vol. 41, No. 3 (2017) 030003*:

$$M(^{234}\text{U}) = 234.0409504 \pm 0.0000024 \text{ g}\cdot\text{mol}^{-1} (k = 2)$$

$$M(^{235}\text{U}) = 235.0439282 \pm 0.0000024 \text{ g}\cdot\text{mol}^{-1} (k = 2)$$

$$M(^{236}\text{U}) = 236.0455662 \pm 0.0000024 \text{ g}\cdot\text{mol}^{-1} (k = 2)$$

$$M(^{238}\text{U}) = 238.0507870 \pm 0.0000032 \text{ g}\cdot\text{mol}^{-1} (k = 2)$$

1 Introduction

1.1 Background

Environmental samples are used by national and international safeguards authorities to verify the absence of undeclared nuclear material and/or activities in nuclear facilities under safeguards [4, 5]. Nuclear security authorities also use environmental samples for evidence collection in cases of border interdictions of nuclear material, and seizures or discoveries of nuclear material in the public domain [6]. Environmental sampling makes use of pieces of cotton cloth, called swipes, to wipe surfaces of interest. The dust collected on these swipes may contain micrometer-sized particles of actinides or other elements of interest. The majority of environmental samples collected for safeguards purposes are taken from bulk handling facilities at the front end of the nuclear fuel cycle (e.g., uranium conversion plants, uranium enrichment plants and uranium fuel fabrication plants), therefore uranium particles are the most often investigated type of particle. In addition to the ^{235}U abundance in uranium particles, the minor isotope abundance ratios $n(^{234}\text{U})/n(^{238}\text{U})$ and $n(^{236}\text{U})/n(^{238}\text{U})$ of uranium in particles may provide additional information to safeguards authorities about equipment or plant design and irradiation history, and may also help to evaluate mixing and decay scenarios. Major and minor uranium isotope ratios in environmental samples collected by inspectors are measured by the IAEA's Office of Safeguards Analytical Services (SGAS) in Seibersdorf, Austria and the IAEA's Network of Analytical Laboratories (NWAL) [7].

Considering the potential consequences of particle analyses in nuclear safeguards, these measurements are subjected to a rigorous quality management system. The reliability and comparability of measurement results of isotope ratios in uranium particles need to be guaranteed and monitored via the correct use of reference materials and quality tools. Laboratories involved in particle analysis have a continuous need for quality control reference materials at the particle level [8].

Therefore, a collaborative project was launched involving the European Commission's Joint Research Centre, Unit G.2 (JRC-Geel, Belgium), the IAEA-SGAS (Austria) and the Forschungszentrum Jülich Institute for Nuclear Waste Management and Reactor Safety (FZJ/IEK-6, Jülich GmbH, Germany) to produce monodisperse, spherical micrometre-sized uranium oxide particles.

One of the batches of monodispersed micrometre-sized uranium particles was selected for certification at JRC-Geel according to the ISO 17034:2016 [1] and ISO Guide 35:2006 [2], and is the subject of this report.

Due to the narrow particle size distribution and the technique used to produce the spherical particles (spray-pyrolysis of a monodisperse aerosol generated using a vibrating orifice aerosol generator), each particle contains approximately the same amount of uranium. Therefore, this material was selected as a suitable candidate for certification for uranium amount per particle according to the ISO 17034 and ISO Guide 35. Prior to its release, the candidate reference material IRMM-2329P was the test item in the NUSIMEP-9 Nuclear Signatures Inter-laboratory Measurement Evaluation Programme [9].

1.2 Choice of the material

Following recommendations from the IAEA regarding the preferred isotopic composition for particle reference materials, suitable uranium solutions were prepared at JRC-G.2 in Geel. This was achieved at JRC-G.2 by mixing two solutions of low-enriched uranium (LEU) material derived from uranium hexafluoride (UF_6) from the JRC-G.2 stock of UF_6 materials. For IRMM-2329P, two solutions derived from IRMM-023 and IRMM-029 were mixed. The mixing of the solutions was not performed strictly using the classical gravimetric approach for which the accurate knowledge of the uranium concentrations and mixing proportions between the two solutions would have to be known. The mixing proportions were only based on estimates from the concentrations and mixing proportions. The isotopic composition of the base solution mixture, called IRMM-2329, was characterised by TIMS using the standardised MTE (modified total evaporation) method [10, 11] and subsequently verified by an external laboratory (IAEA-SGAS) using two different techniques, TIMS/MTE and MC-ICPMS.

The candidate reference material for certification, called IRMM-2329P, consists of spherical uranium oxide micro particles with a mean particle diameter in the micrometre-size range. The particles are characterised by

a high degree of sphericity and a narrow particle size distribution. Such particles allow for better control of the uranium amount per particle, which is one of the certified property values of IRMM-2329P.

The average diameter in the micrometre-sized range was selected to reflect typical samples collected on nuclear safeguards swipe samples [8]. The particles are easily measurable by the current state of practice particle mass spectrometric techniques but also provide a challenge for state-of-the-art techniques to further reduce the measurement uncertainty.

The particles are distributed onto glass-like carbon disks, which allows the samples to be measured directly by various techniques (e.g. SIMS) but also allows the easy transfer of particles onto different substrates (e.g. by micromanipulation). The particles consist of triuranium octoxide (U₃O₈) which ensures that the chemical properties of the particles remain stable over a long period of time [12, 13].

Finally, the uranium isotope amount ratios in IRMM-2329P represent low-enriched uranium (LEU), with a $n(^{236}\text{U})/n(^{238}\text{U})$ ratio, that can be measured by the commonly applied techniques.

1.3 Design of the CRM project

The IRMM-2329P material has been produced, and certified for the uranium isotope amount ratios $n(^{234}\text{U})/n(^{238}\text{U})$, $n(^{235}\text{U})/n(^{238}\text{U})$ and $n(^{236}\text{U})/n(^{238}\text{U})$.

The IRMM-2329P uranium micro particles were produced at the Forschungszentrum Jülich GmbH from uranium solutions, prepared at JRC-Geel G.2 by mixing uranium solutions derived from UF₆ certified reference materials.

Based on evaluation and control of environmental conditions and production processes, no change of the uranium isotope amount ratios from the solutions to the particles is expected during the production. However, the uranium isotope amount ratios in the base solution (mixture) called IRMM-2329 were first measured for characterisation at JRC- G.2 using TIMS/MTE. Then, the isotopic composition was verified at IAEA-SGAS by TIMS/MTE and MC-ICPMS. Subsequently, the integrity of the uranium isotope amount ratios in the produced particles was verified by process control measurements using MC-ICPMS performed at the IAEA-SGAS.

In addition to the uranium isotope amount ratios, the uranium amount per particle in IRMM-2329P was also certified. The uranium amount per particle was determined at JRC-G.2 by isotope dilution with TIMS (ID-TIMS), utilising the ²³³U spike IRMM-058 to establish traceability to the SI. Additionally, the uranium amount per particle was externally verified by IDMS using MC-ICPMS at the IAEA-SGAS.

2 Participants

Project management and evaluation, processing of base solution, homogeneity study, stability study and characterisation have been performed at the European Commission, Joint Research Centre, Directorate G – Nuclear safety and Security, G.2 - Standards for Nuclear Safety, Security and Safeguards in Geel, Belgium.

The production of the particles and the preparation of particles on the glass-like carbon disks (processing) was performed at Forschungszentrum Jülich GmbH, Institute for Energy and Climate Research, IEK-6: Nuclear Waste Management and Reactor Safety in Jülich, Germany.

The verification measurements in the context of the IRMM-2329P certification, for the uranium isotope amount ratios in the base solution and in the particles and the uranium amount per particle, were performed at the International Atomic Energy Agency, Office of Safeguards Analytical Services, Nuclear Material Laboratory and Environmental Sample Laboratory (NML and ESL) in Seibersdorf, Austria.

3 Material processing and process control

3.1 Origin of the starting material

The IRMM-2329P particles were produced from a base solution, the IRMM-2329, which was prepared at the JRC-G.2 by mixing solutions obtained from IRMM-023 (Annex 1) and IRMM-029 (Annex 2), which are two solutions with certified uranium isotope amount ratios. The IRMM-2329 solution was characterised for the isotopic composition using TIMS/MTE [10, 11] and a measurement certificate was issued (Annex 3). The uncertainties of the certified ratios of IRMM-2329 include an additional component for the maximum possible inhomogeneity between different units of the material. This was already introduced and calculated for the certification of the uranium hexafluoride reference materials series IRMM-019-029, which was also performed using TIMS on uranyl nitrate solutions [14, 15].

In order to comply with the ISO 17034:2016, for the certification of the isotopic composition of the IRMM-2329 solution, external verification measurements were performed at the IAEA-SGAS using TIMS/MTE and MC-ICPMS. As shown in Table 1, the results were in good agreement with the values on the measurement certificate, which can therefore be used as certified values for the IRMM-2329 solution. The verification measurement results are compared with the certified values in Table 1.

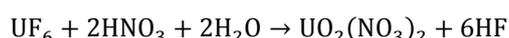
Table 1. Results with expanded uncertainty ($k=2$) of the certified isotope ratios for IRMM-2329 solution and results of verification measurements performed by the IAEA-SGAS.

		$n(^{234}\text{U})/n(^{238}\text{U})$	$n(^{235}\text{U})/n(^{238}\text{U})$	$n(^{236}\text{U})/n(^{238}\text{U})$
Certification Measurement by JRC-G.2	TIMS/MTE (using IRMM-074, Annex 4, for mass bias correction)	0.00034083(19)	0.033902(12)	0.00003021(12)
Verification Measurements by IAEA-SGAS	TIMS/MTE (using IRMM-184, Annex 5, for mass bias correction), by IAEA	0.00034075(41)	0.033894(13)	0.00003019(33)
	MC-ICPMS (1ppb sample solution, using IRMM-025, Annex 6, for mass bias correction)	0.0003411(29)	0.033904(24)	0.00003025(22)

3.2 Processing

The flow scheme for the production of IRMM-2329P is as follows in Figure 1:

The IRMM-2329 base solution was prepared by mixing of IRMM-023 with IRMM-029 as described above. All of the base materials are uranium hexafluoride (UF_6), which were converted to uranyl nitrate ($\text{UO}_2(\text{NO}_3)_2$) by hydrolysis of the gaseous UF_6 in diluted nitric acid, i.e.



The $n(\text{NO}_3)/n(\text{U})$ ratio of the solution was adjusted to around 10 using nitric acid (Suprapur[®]), which was required to convert the solutions to particles [13].

The prepared IRMM-2329 solution was shipped to FZJ, where the solutions were used to produce micrometre-sized triuranium octoxide particles. Detailed information about the particle production process is given in [12, 13 and 16].

The solution was first diluted with ultra-pure water (18.2 MΩ.cm) and ethanol (Ethanol absolute for analysis EMSURE[®], Merck KGaA, Germany) to a uranium content of around 125 $\mu\text{g}\cdot\text{g}^{-1}$ and a water to ethanol volume

ratio around 1. This prepared solution was then fed into a vibrating orifice aerosol generator (VOAG, model 3450, TSI Inc., USA) at a volume flow rate of $2.59 \mu\text{L}\cdot\text{s}^{-1}$. The VOAG was operated using a gold-coated orifice with a diameter of $20 \mu\text{m}$ oscillating with a frequency of 69 kHz. Under the given conditions, a liquid jet was formed, which broke up into monodisperse droplets.

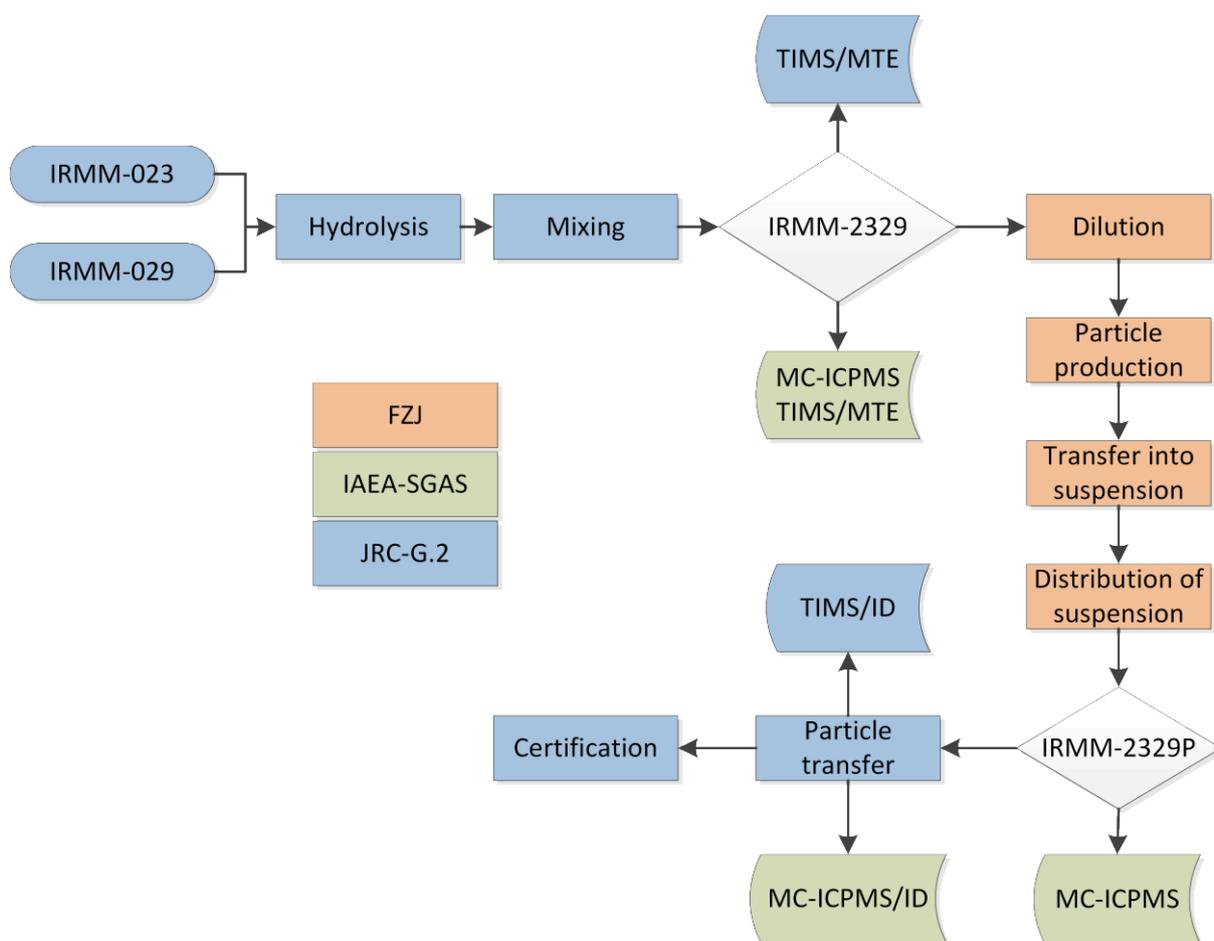
The generated droplets were carried with a pre-cleaned supply of compressed air through a drying column and a 200 mm long aerosol heater (Pressurized air heater, Dekati Ltd., Finland). The solvents (water and ethanol) within the droplets evaporated and resulted into formation of uranyl nitrate precursor particles. The aerosol heater was set to $500 \text{ }^\circ\text{C}$, at which temperature the uranyl nitrate precursor particles were decomposed and partially reduced to solid triuranium octoxide micro particles.

After passing through the aerosol heater, the particle-bearing flow was passed through a 500 mm long air-cooled stretch. The particles were then extracted from the particle-bearing stream by means of a single-stage inertial impactor onto a 25.4 mm (1 inch) diameter quartz disk. For each material, 14 quartz disks were produced over a course of two subsequent days.

The quartz disks were transferred into an ethanol-filled bottle, which was ultra-sonicated to detach particles from the quartz disks into the ethanol. The stability of the produced particles in an ethanol suspension was previously demonstrated [13, 17].

Aliquots of the produced suspension were distributed by pipette onto 25 mm diameter glass-like carbon disks, which were gently heated to dryness to homogeneously deposit the particles onto the substrate. Before packing, the disks were shortly heated using a heating plate at $350 \text{ }^\circ\text{C}$ to evaporate all volatile residues.

Figure 1. Flow-scheme of the production of uranium micro particles IRMM-2329P from the two IRMM certified reference materials with the certification and verification measurements related to the uranium isotope amount ratios and the uranium amount per particle.



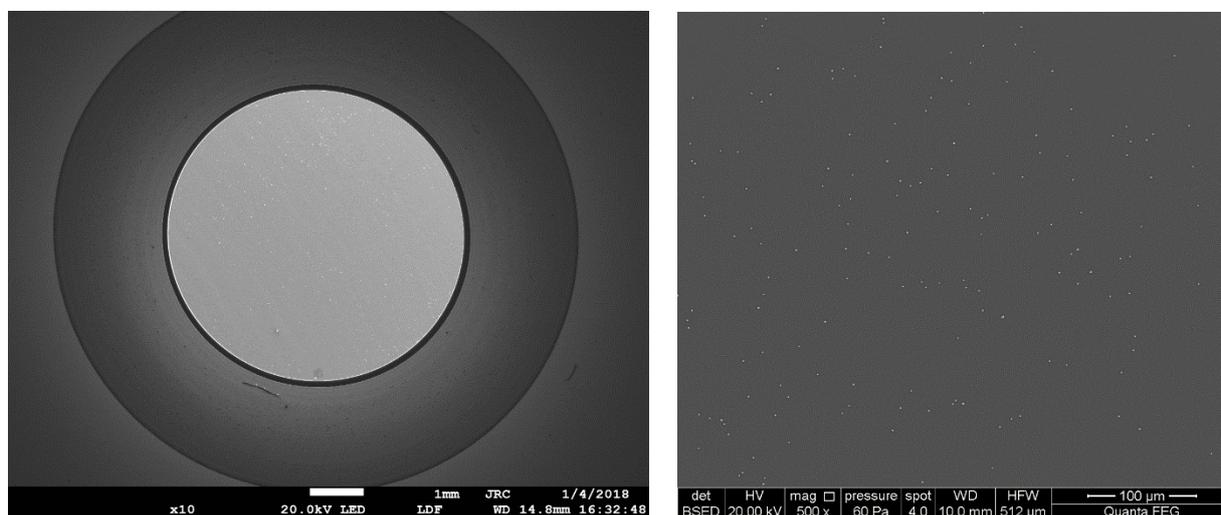
Finally, 110 units of IRMM-2329P were produced using the procedure described above. An IRMM-2329P unit consists of ca. 15000 monodisperse uranium particles of micrometre-sized diameter, distributed onto a 25 mm diameter glass-like carbon disk.

3.3 Process control of the IRMM-2329P particles production

Prior to the production of the particles from the diluted solutions, a test run was performed at FZJ using the intended solution and parameters. The produced particles were examined by using a scanning electron microscope (SEM). Once the presence of spherical particles had been confirmed, the particle production started. During the production, an optical particle sizer (OPS, model 1110, TSI Inc., USA) provided an online measurement of the (estimated) particle count and particle size distribution.

The final produced particles were investigated by SEM before dispersion over the glass-like carbon disks and after dispersion, a number of randomly selected samples were investigated by SEM to provide an estimate of the particle morphology and size distribution and the number of particles present on the samples.

Figure 2. SEM images of a carbon planchet covered with IRMM-2329P particles. The picture on the left shows the full glass-like carbon planchet (magnification x10). The right picture (taken at FZJ) gives a more detailed view of the uranium particles present (magnification x500).



Based on these studies, it was found that the units of IRMM-2329P contained approximately 15000 particles per unit. Although quantification of the particle size proved difficult, the particle diameters was found to range between 1 and 2 μm with a relative standard deviation of the most frequented population of about 3.4 % (indicative values for particle diameter and standard deviation, not certified).

In addition to the main particle population, approximately 4 % of the particles contained double the amount of uranium compared to the main particle population (see section 4.2.2). Such particles are likely produced by the fusion of two droplets or particles during the particle production [12].

4 Homogeneity

A key requirement for any reference material aliquoted into units is equivalence between those units. In this respect, it is relevant whether the variation between units is significant compared to the uncertainty of the certified value, but it is not relevant if this variation between units is significant compared to the analytical variation. Consequently, ISO 17034 [1] requires reference material (RM) producers to quantify the between-unit variation. This aspect is covered in between-unit homogeneity studies.

The within-unit inhomogeneity does not influence the uncertainty of the certified value when the minimum sample intake is respected, but determines the minimum size of an aliquot that is representative for the whole unit. Quantification of within-unit inhomogeneity is therefore necessary to determine the minimum sample intake.

4.1 Between-unit homogeneity

The between-unit homogeneity was evaluated to ensure that the certified values of the IRMM-2329P are valid for all produced units of the material, within the stated uncertainties.

4.1.1 Uranium isotopic composition

As shown below in section 6.1 about the characterisation and process control measurements, the isotopic composition of the particles was verified to be in agreement with the certified composition of the solution. Therefore, no between-unit homogeneity study for the uranium isotopic composition of the particles was deemed necessary.

4.1.2 Uranium amount per particle

The between-unit homogeneity study for the uranium amount as derived from the mean uranium amount content of 10 dissolved particles in IRMM-2329P was performed on a set of six units with the numbers 16, 20, 51, 69, 78, 102, which were selected from a random stratified sampling scheme. The number of units corresponds to the approximate cubic root of the total number of produced test items (110 units), rounded up and increased by 1, as the units were also intended to be used for the characterisation studies.

From unit no. 16 fifty particles were transferred onto five different carburised single Rhenium filaments (10 particles per filament), using micromanipulators installed under an optical microscope. The same particle transfer was also applied from the units no. 20, 51, 69, 78 and 102. This resulted in five aliquots per unit and 30 filaments with 10 particles on each filament.

After transfer, 3 μL of a ^{233}U spike (IRMM-058, Annex 7) was added volumetrically using a calibrated pipette (Annex 8). Then, 2 μL of 3 $\text{mol}\cdot\text{L}^{-1}$ HNO_3 (Suprapur[®]) was added in order to dissolve the particles and the filament was heated using a current of 0.4 A to evaporate the solvent.

The measurements of the filaments were performed by TIMS (Triton), during which ^{238}U , ^{235}U and ^{233}U were measured subsequently using the ion counter detector. The degree of homogeneity between the uranium from the spike and from the particles was assessed by observation of the $n(^{233}\text{U})/n(^{238}\text{U})$ ratio during the course of the measurement. In case of significant changes or drifts of the $n(^{233}\text{U})/n(^{238}\text{U})$ ratio during the measurements the results of that measurement were excluded from the overall evaluation. Due to the limited number of positions of a turret, the prepared aliquots were divided over three turrets and were therefore analysed under intermediate precision conditions. For each turret a procedural blank was measured. Additionally, four quality control samples prepared from IRMM-023 micro particles were analysed.

The measurements were performed in a randomized manner to detect possible trends in the analytical and unit production sequences.

The amount of uranium present on the filament (n_x) was calculated using the GUM Workbench software¹ and applying the IDMS equation (Equation 1). The complete IDMS results for the uranium amount of ten dissolved particles per filament in IRMM-2329P are presented in Annex 9.

$$n_x = \frac{R_y - R_b}{R_b - R_x} \cdot \frac{\Sigma R_x}{\Sigma R_y} \cdot n_y \quad \text{Equation 1}$$

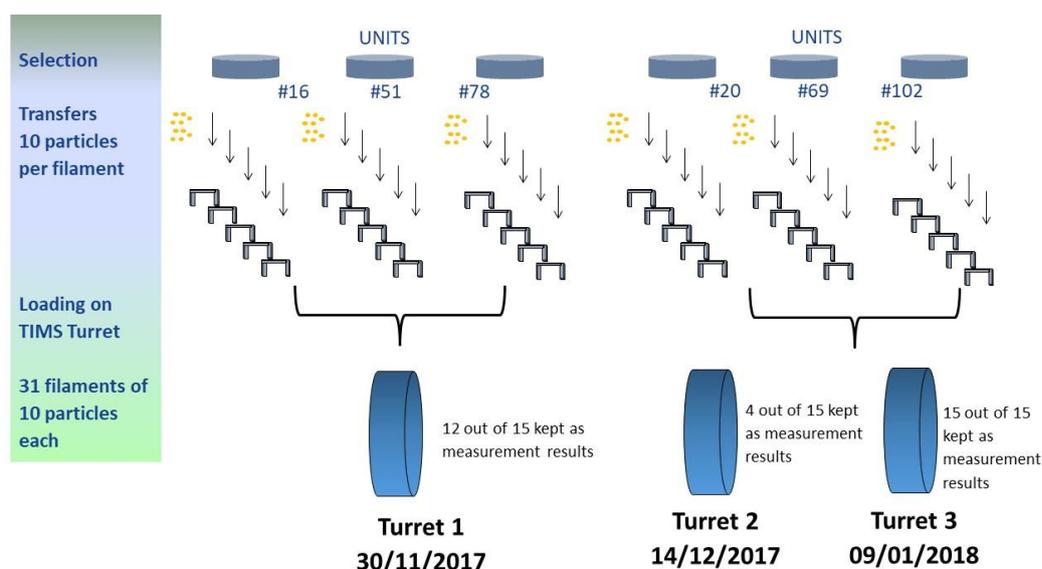
in which R_y , R_x and R_b are the $n(^{233}\text{U})/n(^{238}\text{U})$ isotope amount ratios of the spike, sample and blend respectively. The isotope amount ratio of the spike (IRMM-058) was taken from the certificate (Annex 7), the isotope amount ratio of the sample was taken from the certified value of the IRMM-2329P particles (section 7.1, Annex 3) and the isotope amount ratio of the blend was measured by TIMS (Annex 9). ΣR_x and ΣR_y are the sums of isotope amount ratios of the sample and spike respectively and n_y is the amount of uranium added to the blend, which was calculated based on the certified isotope amount content, the transferred volume and the density of the spike, calculated in accordance to Sakurai and Tachimori [18].

The uranium amount of ten dissolved particles per filament was calculated by subtracting the amount of uranium measured in the procedural blank analysed with each TIMS turret (Annex 9). Finally the calculated uranium amount in each aliquot was divided by the number of transferred particles, (Annex 9) to determine the uranium amount per particle.

Due to an incomplete mixing of the spike with the sample on individual filaments, indicated by significant changes of the measured $n(^{233}\text{U})/n(^{238}\text{U})$ ratios during the TIMS measurements (where ^{233}U is originating from IRMM-058 spike and ^{238}U mainly from IRMM-2329P particles), 14 of the prepared aliquots were rejected. In fact, almost a complete turret of results was rejected and a new turret for units no 20, 69 and 102 was prepared (see Figure 3), resulting in a total of 31 IDMS measurements.

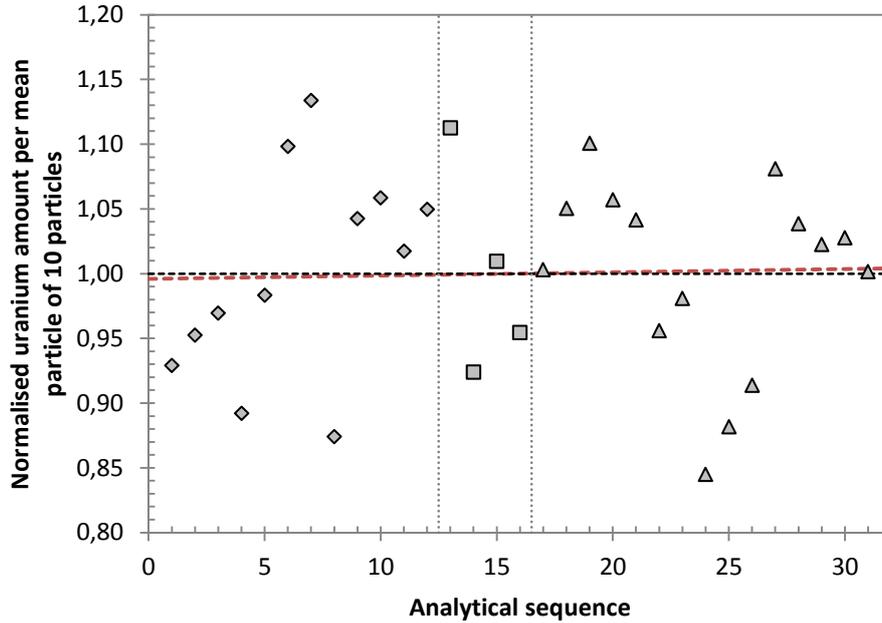
The analyses were performed on three turrets, and therefore under intermediate measurement precision conditions. To exclude any intermediate precision effects on the between-unit homogeneity, the obtained results were normalised to the averages of the respective turrets. The normalised data did not show any significant ($p = 95\%$) trends in either the order of analysis (Figure 4) or the order of production. Using Grubb's test, no significant ($p = 95\%$) outliers were detected in any of the analytical results or the unit means. Also, the obtained results show a normal distribution, based on visual inspection of quantile-quantile plots and prepared histograms.

Figure 3. Aliquoting of 10 particles per filament from units 16, 20, 51, 69, 78, 102 onto 30 filaments



¹ GUM Workbench, Programme GUM workbench 2.4, Metrodata GmbH, 2009, Weil am Rhein, Germany

Figure 4. Normalised uranium amount as derived from the mean uranium amount content of 10 dissolved particles (IRMM-2329P) in analytical sequence with their average (black line) and linear regression line (red line). The different symbols represent the different three turrets.



Quantification of between-unit homogeneity was performed by analysis of variance (ANOVA), which separates the between-unit variation (s_{bb}) from the within-unit variation (s_{wb}). The latter is equivalent to the combined method intermediate precision and the possible within-unit inhomogeneity (i.e. here within the planchet).

Evaluation by ANOVA requires mean values per unit, which follow at least a unimodal distribution and results for each unit that follow unimodal distributions with approximately the same standard deviations. The distribution of the mean values per unit was visually tested using histograms and normal probability plots. Too few data are available for the unit means to make a clear statement of the distribution. Therefore, it was checked visually whether all individual data follow a unimodal distribution using histograms and normal probability plots. Only minor deviations from unimodality of the individual values were observed, and do not significantly affect the estimate of between-unit standard deviations.

It should be noted that s_{bb} and s_{wb} are estimates of the true standard deviations and are therefore subject to random fluctuations. Therefore, the mean square between units ($MS_{between}$) can be smaller than the mean squares within units (MS_{within}), resulting in negative arguments under the square root used for the estimation of the between-unit variation, whereas the true variation cannot be lower than zero. In this case, u_{bb}^* , the maximum inhomogeneity that could be hidden by method repeatability, was calculated as described by Linsinger et al. [19, 20]. u_{bb}^* is comparable to the limit of detection of an analytical method, yielding the maximum inhomogeneity that might be undetected by the given study setup. Method intermediate precision (s_{wb}), between-unit standard deviation (s_{bb}) and u_{bb}^* were calculated as:

$$s_{wb} = \sqrt{MS_{within}} \quad \text{Equation 2}$$

$$s_{bb} = \sqrt{\frac{MS_{between} - MS_{within}}{n_0}} \quad \text{Equation 3}$$

$$u_{bb}^* = \sqrt{\frac{MS_{within}}{n_0}} \cdot \sqrt[4]{\frac{2}{v_{within}}} \quad \text{Equation 4}$$

MS_{within} mean of squares within-unit from an ANOVA
 $MS_{between}$ mean of squares between-unit from an ANOVA
 n_0 mean number of replicates per unit

V_{within} degrees of freedom of MS_{within}

The results of the measurements are shown in Annex 10. The results of the evaluation of the between-unit variation are summarised in Table 2. The resulting values from the above equations were converted into relative uncertainties.

Table 2. Results of the between-unit homogeneity study of the uranium amount as derived from the mean uranium amount content of 10 dissolved particles. The quantity $U_{\text{hom,rel}}$ is calculated by combining $S_{\text{wb,rel}}$ and $S_{\text{bb,rel}}$.

	$S_{\text{wb,rel}}$ [%]	$S_{\text{bb,rel}}$ [%]	$U_{\text{bb,rel}}^*$ [%]	$U_{\text{hom,rel}}$ [%]
IRMM-2329P	5.5	5.5	1.3	7.8

The homogeneity study showed no outlying unit means or trends in the production sequence. As U_{bb}^* sets the limits of the study to detect inhomogeneity and the value of S_{bb} is larger than U_{bb}^* , S_{bb} is adopted as uncertainty contribution to account for potential between-unit inhomogeneity. Finally, the within-unit standard deviation S_{wb} and the between-unit standard deviation S_{bb} were added in quadrature to calculate the combined uncertainty of the homogeneity study, labelled as U_{hom} .

4.2 Within-unit homogeneity and minimum sample intake

The within-unit homogeneity is closely correlated to the minimum sample intake. Due to this correlation, individual aliquots of a material will not contain the same amount of analyte. The minimum sample intake is the minimum amount of sample that is representative for the whole unit and thus should be used in an analysis. Using sample sizes equal or above the minimum sample intake guarantees the certified value within its stated uncertainty.

4.2.1 Uranium isotopic composition

Based on the thorough evaluation and control of environmental conditions during the production processes, the uranium isotopic composition can be considered being homogeneous throughout the unit and no minimal sample intake needs to be considered.

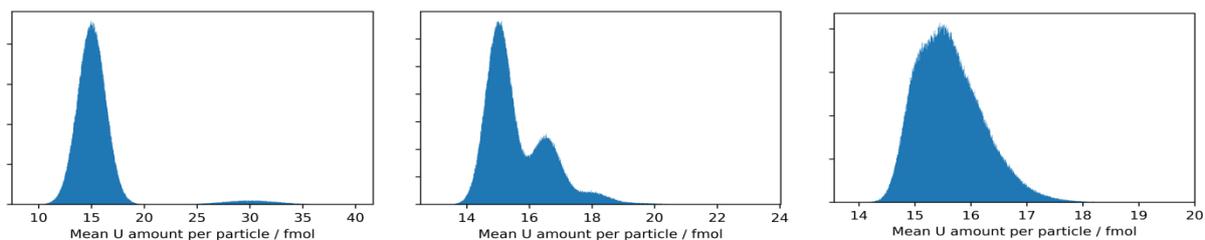
4.2.2 Uranium amount per particle

During the particle production by means of a spray-pyrolysis method, a number of particles are produced containing double the amount of uranium compared to the main particle population, i.e. the certified value [14]. SEM analysis performed at JRC-Geel has shown that about 4.0 % of the IRMM-2329P particles contain double the amount of uranium. The possible selection of this kind of particles is contributing to the within-unit inhomogeneity. Therefore, a minimal number of particles to be analysed needs to be taken into account. Due to the existence of particles containing double the amount of uranium with a sample-size-independent fraction of about 4.0 %, the within-unit inhomogeneity does not diminish even if a very large number of particles is taken as a sample. But a minimal sample intake can be calculated in a way that the within-sample inhomogeneity would have a negligible impact on the (combined) uncertainty of the certified uranium amount per particle.

For the uranium amount as derived from the mean uranium amount content of 10 dissolved particles in IRMM-2329P, the largest uncertainty contribution is given by the uncertainty of the homogeneity study U_{hom} of 7.8%. This would lead to a maximum permissible uncertainty due to the within-unit inhomogeneity $U_{\text{wb,max}}$ of 2.6 % ($k = 1$) and a maximum combined uncertainty of the certified value $U_{\text{CRM,max}}$ of 8.7 % ($k = 1$).

Monte-Carlo simulations were performed on $m = 10^6$ sets of N particles each, where N was varied from 1 to 30. For each set of N particles, the mean uranium amount per particle of the set was calculated, based on a random selection of particles containing either once ($p = 96.0\%$) or twice ($p = 4.0\%$) the amount of uranium, which was taken randomly from a Gaussian distribution with the average of distribution $\mu = 15.1$ fmol (x_{CRM}) and the variance of distribution $\sigma = U_{CRM,max}$. Figure 5 shows the obtained distributions of the mean uranium amount per particle, based on the random selection of N particles with a probability of 4.0 % to select a particle with double the amount of uranium.

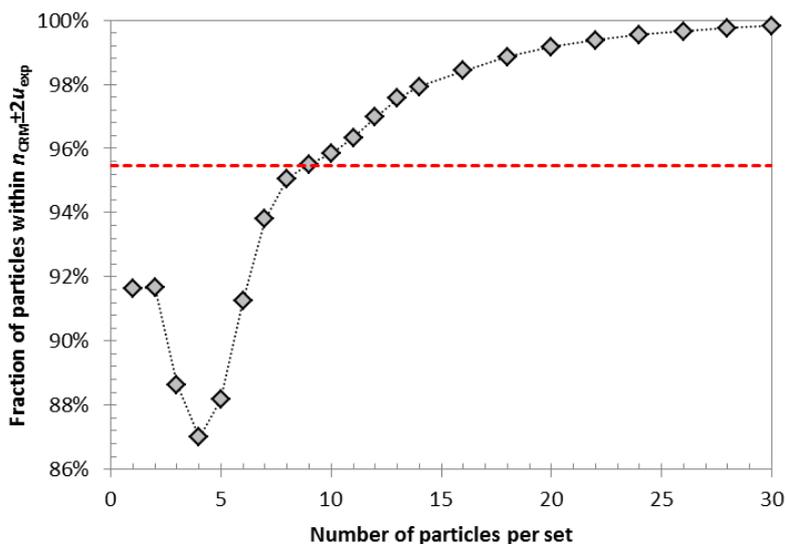
Figure 5. Histograms of 10^6 sets of 1 (left), 10 (centre) and 30 (right) randomly selected particles.



For each simulation consisting of sets of N particles, the fraction of results falling within the $x_{CRM} \pm 2 \cdot U_{CRM,max}$ range was counted, i.e. the fraction of results which would fall within the 95.45 % confidence interval with a negligible effect due to the within-unit inhomogeneity.

Based on the performed simulations, it was found that at least 10 particles need to be selected to obtain results with a negligible impact from the within-unit inhomogeneity. The minimal sample intake for the uranium amount per particle for IRMM-2329P is set to 10 particles, unless the applied analytical method, such as LG-SIMS, FT-TIMS or LA-MC-ICPMS, is capable of either identifying or selectively excluding the particles containing double the amount of uranium. In that case the minimal sample intake would be 1 particle.

Figure 6. Fraction of means within two times the maximum permissible uncertainty including within-unit inhomogeneity from the certified value.



5 Stability

Stability testing is necessary to establish the conditions for storage (long-term stability) as well as the conditions for dispatch of the materials to the customers (short-term stability). During transport, especially in summer time, temperatures up to 60 °C can be reached and stability under these conditions must be demonstrated, if the samples are to be transported without any additional cooling.

5.1 Short-term stability study

5.1.1 Uranium isotope amount ratios

As the uranium isotopic composition is independent of the temperature, there is no impact from the transportation on the uranium isotopic composition. Therefore, no short-term stability study was performed for the uranium isotope amount ratios in the particles.

5.1.2 Uranium amount per particle

Stability studies for the uranium amount per particle, as derived from the mean uranium amount content of 10 dissolved particles of IRMM-2329P, were carried out using an isochronous design. In this approach, two units of IRMM-2329P were selected using a randomly stratified scheme. Before storage, two aliquots (two filaments) were prepared from each unit, after which the unit was stored for a particular length of time at two different temperature conditions. After defined periods of time, the units were shortly removed from the controlled temperature conditions to prepare two more aliquots (two more filaments per unit), before the units were stored back under controlled temperatures.

For the short-term stability study, one unit was stored at 4 °C and the other at 60 °C for 7, 14 and 21 days. Note that at the end of the 21-day storage, three aliquots (three filaments) were prepared instead of two. Finally, the 18 aliquots (18 filaments) were measured by ID-TIMS in a similar way as described in section 4.1.2. At the end of the isochronous storage, the prepared aliquots were analysed simultaneously under repeatability conditions.

The data were evaluated individually for each temperature. The results were screened for outliers using the single and double Grubbs test on a confidence level of 99 %, no outliers were found. In addition, the data were evaluated against storage time, and regression lines of the uranium amount per particle versus time were calculated to test for potential increase/decrease of the uranium amount per particle due to shipping conditions. The slopes of the regression lines were tested for statistical significance. None of the trends was statistically significant at a 95 % confidence level for any of the temperatures. The results of the short-term stability measurements are shown in Annex 11.

As a conclusion, the uncertainty from the short term stability study u_{tm} does only reflect an uncertainty due to a possible inhomogeneity within or between planchets, which is already taken into account by the homogeneity study, as described above. Thus there is no uncertainty contribution to be considered from the short-term stability.

The material can be dispatched without further precautions under ambient conditions.

5.2 Long-term stability study

5.2.1 Uranium isotope amount ratios

Data from the certification and monitoring program for various types of uranium CRMs have demonstrated that the uranium isotopic composition remained stable over a timeframe of 30 years, and the relative uncertainty of the $n(^{235}\text{U})/n(^{238}\text{U})$ isotope amount ratio due to long-term stability for a shelf-life of 10 years can be considered negligible compared to the uncertainty due to characterisation [21]. Also, it is known that all uranium isotopes have the same physical properties, except for the half-life. As the half-lives² of ^{234}U ($245.5 \cdot 10^3$ a), ^{235}U ($704 \cdot 10^6$ a), ^{236}U ($23.43 \cdot 10^6$ a) and ^{238}U ($4.468 \cdot 10^9$ a) are much longer than the intended

² DDEP-BIPM (accessed: 22 February 2018) http://www.nucleide.org/DDEP_WG/DDEPdata.htm

shelf life of the material, no significant effect due to radioactive decay is expected on the uranium isotope amount ratios. It can therefore be concluded that the stability effects on the uranium isotope amount ratios are negligible. The particle reference material IRMM-2329P will also be placed under the JRC-Geel G.2 regular stability monitoring program to further verify the stability.

5.2.2 Uranium amount per particle

Due to the physical properties of uranium, no change of the uranium amount per particle (IRMM-2329P) is to be expected during the storage of the material. It was demonstrated that specific particle production process yields particles consisting of U_3O_8 [12], which is considered to be one of the most stable uranium species [22]. Consequently, no long-term instability of the uranium amount per particle is expected to occur. Also, previous investigations have demonstrated that no degradation of UO_2F_2 micro particles occurred [23, 24], which are expected to have a lower degree of stability compared to U_3O_8 micro particles.

However, after certification, the material will be included in the JRC's regular stability monitoring programme (post-certification monitoring according to ISO Guide 35 [2]), to control its further stability.

6 Characterisation

The material characterisation is the process of determining the property value(s) of a reference material. The material characterisation was based on a primary isotope ratio method of measurement combined with a primary direct method (gravimetry) [25], and confirmed by an independent method. A primary method of measurement (also called "primary reference method" in the International Vocabulary of Metrology (VIM) [26]) is a method that does not require calibration with a standard of the same measurand and does not depend on a chemical reaction³. Such methods are of highest metrological order and often yield results with low uncertainties. However, it is nevertheless prudent to demonstrate absence of bias or gross errors by use of an independent method of lower metrological order.

6.1 Uranium isotope amount ratios in IRMM-2329P

The certification of the isotopic composition of the IRMM-2329 base solution was also applied to the IRMM-2329P particle reference material after so-called "process control measurements" (PCM) had been performed successfully. The PCM measurements were designed in a way to control the entire processing of the base solution, including the particle production using the base solution and also the process of dissolving the particles back into a nitrate solution. The PCM measurements of the uranium isotope amount ratios in the IRMM-2329P particles were performed by MC-ICPMS at IAEA-SGAS. There, the particles were leached from the sample planchets either by complete soaking of the IRMM-2329P planchet in HNO₃ or by adding a drop of HNO₃ to the particle-bearing side of the planchet (drop-leaching).

Three units of IRMM-2329P were analysed using the complete soaking method and three other units using the drop-leaching. The IRMM-025 certified reference material was used as standard for mass fractionation correction and calibration of the ion counters during the MC-ICPMS measurements.

The MC-ICPMS measurements were performed under repeatability conditions. Apparently, small differences were found between the measured $n(^{235}\text{U})/n(^{238}\text{U})$ ratios of the IRMM-2329 base solution and the solutions obtained by leaching the IRMM-2329P particles using the two methods. Such an alteration of the isotope amount ratio could possibly be induced during the particle production, either

1. due to a uranium blank associated with the blank planchet without any particles on it.
2. by a cross-contamination from "foreign" particles on the planchet. This would not have an effect on the isotope amount ratios of individual particles, but only on the isotope amount ratios of the total uranium material on the planchet, for example during leaching experiments.

The two above mentioned sources of possible alterations were excluded by systematic analyses of the surfaces of several planchets. The surfaces of blank planchets were first analysed with the Automated Particle Measurement (APM) screening procedure at the LG-SIMS instrument at the IAEA laboratories, where no uranium background was observed. Secondly, the surfaces of blank planchets were analysed by leaching with HNO₃ with subsequent ICPMS analysis at FZJ, and also using this technique no uranium was found on the surfaces of the planchet.

Furthermore, the APM screening procedure at the LG-SIMS instrument at the IAEA was applied to screen planchets with IRMM-2329P particles on them. During the analyses no "foreign" particles with ²³⁵U enrichments other than the main population, which enrichment is in agreement with the base solution, were found.

After firstly excluding a possible contamination of the blank planchet prior to the particle production using two different methods, and secondly also excluding a cross-contamination with "foreign" particles on the planchet, no further explanation was found for the observed small differences for the measured $n(^{235}\text{U})/n(^{238}\text{U})$ ratios from the base solution.

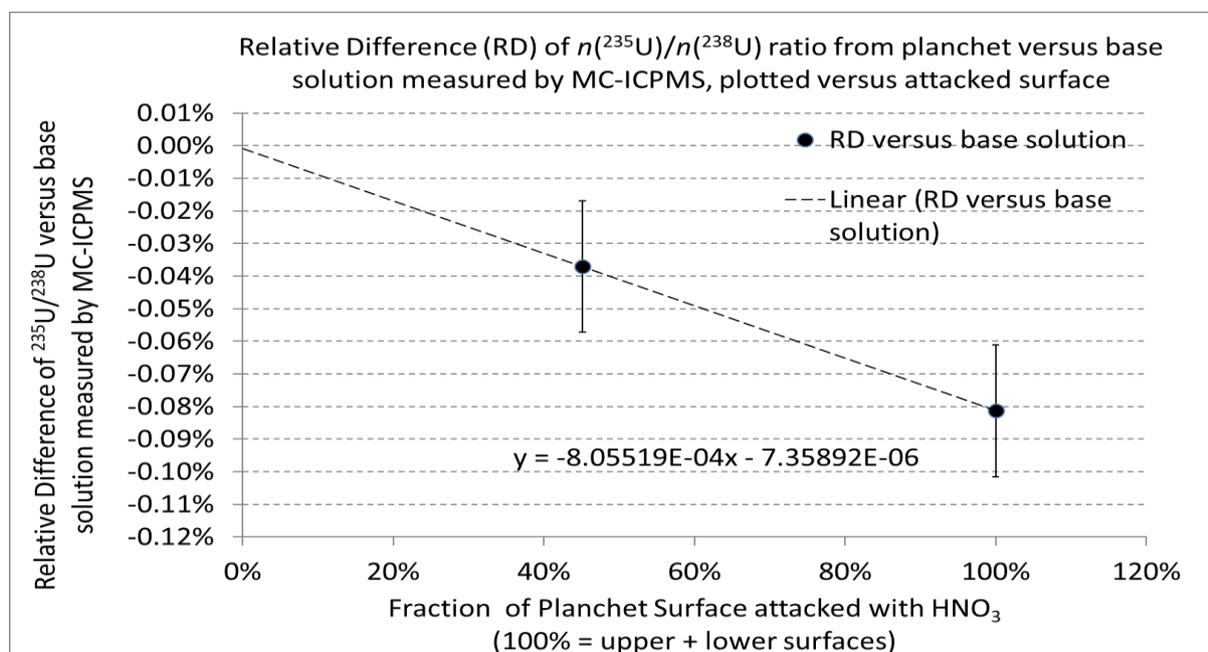
On the other hand, the leaching method introduced here for the purpose of the process control measurements of the isotope ratios, in combination with high sensitive and high precision MC-ICPMS measurements of small size samples (superior compared to TIMS), is not a method that will be applied by the users of this particle

³ A primary method is "a method having the highest metrological qualities, whose operation(s) can be completely described and understood and for which a complete uncertainty statement can be written in terms of SI units. A primary ratio method measures the value of a ratio of an unknown to a standard of the same quantity; its operation must be completely described by a measurement equation

reference material. This method was chosen specifically for this project in order to achieve high precision verification measurements of the isotope ratios of IRMM-2329P using a mass spectrometric technique, which is independent on the typical particle mass spectrometric techniques such as SIMS, FT-TIMS and LA-MC-ICPMS. These are the techniques, which will be applied by the customer and which will then be calibrated using this new particle reference material. But as a consequence of the observed differences, special handling instructions have to be given, as explained below.

The results of the MC-ICPMS-measurements on the leached particles are presented in detail in Figure 7. It shows the relative differences of the measured $n(^{235}\text{U})/n(^{238}\text{U})$ isotope ratios of the two IRMM-2329 solutions, which were obtained by leaching IRMM-2329P particles from the planchets by attacking with HNO_3 , by either 100% ("complete soaking") or 45% ("drop leaching") of the surface, which are given relative to the $^{235}\text{U}/^{238}\text{U}$ isotope ratios of the base solution measured using the same technique, in this case MC-ICPMS. These differences are plotted versus the fraction of the planchet surface which was attacked by the HNO_3 acid for leaching for the two methods. The results indicate a bias of the $n(^{235}\text{U})/n(^{238}\text{U})$ ratio from the samples which were entirely soaked (100% of the sample), which is approximately twice as large as the bias measured with the drop-leaching technique (ca. 45% of the surface covered with HNO_3). Therefore the bias is proportional to the surface attacked with nitric acid.

Figure 7. Relative Difference of measured $n(^{235}\text{U})/n(^{238}\text{U})$ isotope amount ratio of the IRMM-2329 solutions, which were obtained by leaching IRMM-2329P particles from the planchets, from the ratios of the base solution measured also by MC-ICPMS. The error bars are given as combined standard errors, they do not include common uncertainty components related to the mass bias correction and reference materials used for it.



The extrapolation of the relative difference in the $n(^{235}\text{U})/n(^{238}\text{U})$ isotope ratios versus the attacked surface towards the surface actually covered by the about 15000 particles with about 1.3 μm diameter per particle on the planchet - which is about 0.002% relative to the total surface - leads to an extrapolated relative difference for the $n(^{235}\text{U})/n(^{238}\text{U})$ isotope ratios of $(0.00074 \pm 0.029)\%$, which is negligible. The extrapolation yields a negligible difference of $(0.0012 \pm 0.029)\%$ even in the case, that a square $20\mu\text{m} \times 20\mu\text{m}$ area is taken into account for each particle, which represents an upper limit for the surface area typically rastered with the primary beam during microbeam measurements by LG-SIMS.

The extrapolation to the hypothetical case that only the total particle's surface area (or the area rastered during SIMS analyses) and none of the impurities of the surrounding surface were dissolved by the acid, is therefore leading to the conclusion that the $n(^{235}\text{U})/n(^{238}\text{U})$ isotope ratio within the leached particles is in agreement with the values for the base solution measured with the same technique (MC-ICPMS, see Table 3).

This was already found to be in agreement with the certified ratio for the base solution (see Table 1), which can therefore be used for the certification of the particles.

Table 3: Results of process control measurements by MC-ICPMS at the IAEA-SGAS. The extrapolated major ratio $n(^{235}\text{U})/n(^{238}\text{U})$ is marked (*). For the minor ratios minor isotope ratios $n(^{234}\text{U})/n(^{238}\text{U})$ and $n(^{236}\text{U})/n(^{238}\text{U})$ no extrapolations had to be performed (**), see text below for further explanations.

		$n(^{234}\text{U})/n(^{238}\text{U})$	$n(^{235}\text{U})/n(^{238}\text{U})$	$n(^{236}\text{U})/n(^{238}\text{U})$
Complete soaking (100% surface leached)	Leached Particles using MC-ICPMS (1ppb sample solution, calibrated using IRMM-025)	0.0003403(18)	0.033879(10) (-0.081% vs base solution)	0.00003020(15)
Drop leaching (45% surface leached)	Leached Particles using MC-ICPMS (1ppb sample solution, calibrated using IRMM-025)	0.0003405(20)	0.033894(10) (-0.037% vs base solution)	0.00003020(19)
Extrapolated	Leached Particles using MC-ICPMS (1ppb sample solution, calibrated using IRMM-025)	(**)	0.033906(13) (*)	(**)
Base solution for comparison	Base solution by MC-ICPMS (1ppb sample solution, calibrated using IRMM-025)	0.0003411(29)	0.033907(10)	0.00003025(22)

The process control measurements of the minor isotope ratios $n(^{234}\text{U})/n(^{238}\text{U})$ and $n(^{236}\text{U})/n(^{238}\text{U})$ for the leached particles did not show any significant relative differences relative to the base solution, which is due to the much larger measurement uncertainties compared to the so-called "major" ratios $n(^{235}\text{U})/n(^{238}\text{U})$. Due to the very close agreement for the minor isotope ratios with the base solution values no extrapolation had to be performed. As a conclusion, the major ratios $n(^{235}\text{U})/n(^{238}\text{U})$ as well as the minor ratios $n(^{234}\text{U})/n(^{238}\text{U})$ and $n(^{236}\text{U})/n(^{238}\text{U})$ obtained by the process control measurements are in agreement with the certified values for the base solution. The results of the process control measurements by MC-ICPMS at the IAEA-SGAS are summarised in Table 3.

The employed method for the process control measurements by leaching particles from the planchets with subsequent MC-ICPMS measurements was especially designed for the verification of the isotope ratios within the IRMM-2329P particles. This method does not represent the typical way of handling or using this particle reference material. Following the observations of the process control measurements, the use of the IRMM-2329P particle reference material for isotope ratio analyses is restricted to the application of particle measurement techniques, such as LG-SIMS, FT-TIMS, TIMS with micro-manipulation and LA-MC-ICPMS, which only affect a maximum surface area of $20\mu\text{m} \times 20\mu\text{m}$ around the particles to be analysed.

6.2 Uranium amount per particle

The determination of the uranium amount per particle in IRMM-2329P was based on isotope dilution mass spectrometry (IDMS) using the IRMM-058 spike (^{233}U) carried out at JRC-Geel G.2 and externally verified by the IAEA-SGAS Environmental Samples Laboratory, using CRM995 and CRM111-A and ID-MC-ICPMS.

6.2.1 Determination measurements by ID-TIMS

A detailed description of the analyses is given in section 4.1.2, as the characterisation of the material was based on the analyses performed for the homogeneity assessment.

The complete ID-TIMS results and calculations using the software GUM Workbench for the characterisation study are presented in Annex 9. Based on the 31 replicate measurement data performed by ID-TIMS for the homogeneity study, a mean uranium amount per particle of 15.052 fmol, i.e. 10^{-15} mol (rounded to 15.1 fmol, equivalent to a mass of 3.58 pg) was calculated. Full uncertainty budgets were established in accordance with the 'Guide to the Expression of Uncertainty in Measurement' [3].

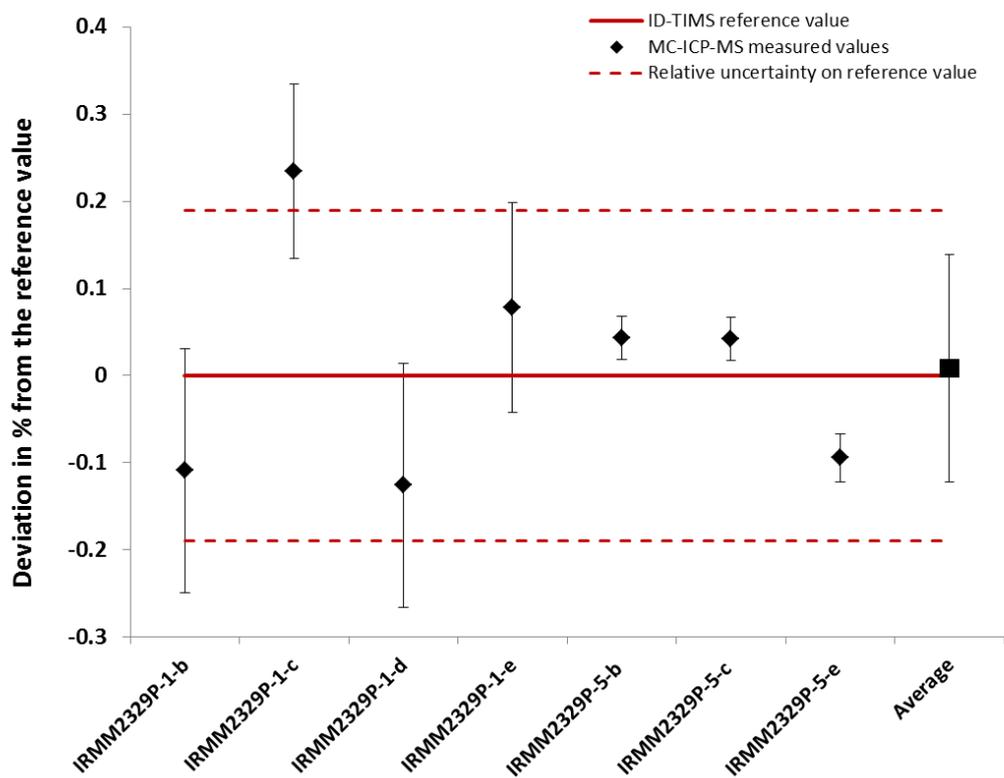
As the replicate measurements were performed under intermediate precision conditions, the relative uncertainty of the mean uranium amount per particle (0.053 %) was combined with the relative intermediate precision uncertainty (4.0 %), which mainly consists of the uncertainty of the transferred volume and the density of the spike (Annex 9). The combined standard uncertainty for the characterisation is therefore 0.595 fmol (or 0.14 pg) ($k = 1$), which corresponds to a relative combined standard uncertainty of 4.0 %.

6.2.2 Confirmation measurements ID-MC-ICPMS

The uranium amount per particle in IRMM-2329P as determined by ID-TIMS was confirmed by external verification measurements performed at the IAEA-SGAS using ID-MC-ICPMS (8). These measurements were carried out on 10 sub-samples from an IRMM-2329P unit, provided by JRC-Geel G.2, and consisting of 5 aliquots containing a single U particle each and 5 aliquots containing 5 particles each. This procedure was especially designed for MC-ICPMS measurements at IAEA-SGAS. Once at IAEA-SGAS, the samples were first screened for any potential contamination and to determine the amount of ^{233}U spike to be used, using one sample with a single U particle and one with 5 U particles. Then ID-MC-ICPMS measurements were performed on the 8 remaining sub-samples/aliquots using uranium spikes at IAEA-SGAS (NBS995 and CRM-111A). Procedural blank samples were also measured together with the U particle sub-samples and the U IDMS measurement results were corrected for the blank contribution. A full uncertainty budget was established in accordance with the 'Guide to the Expression of Uncertainty in Measurement' [3].

Measurement results for U amount per particle in IRMM-2329P using MC-ICPMS are presented in Figure 8. Finally, only 7 sub-samples were used for the IDMS calculations, since one measurement result with 5 U particles was considered as outlier.

Figure 8 Verification measurement results of the U amount per particle using ID-MC-ICPMS compared to the ID-TIMS reference value and its final uncertainty ($k = 2$) as given in Table 6



7 Value assignment

Certified values are values that fulfil the highest standards of accuracy.

The assigned uncertainties consist of uncertainties relating to characterisation, u_{char} (section 6), potential within-unit and between-unit inhomogeneities, combined and expressed as u_{hom} (section 4.1), potential degradation during transport, u_{trn} (section 5.1), and potential degradation during storage, u_{sts} (section 5.2). These different contributions were combined to estimate the relative expanded uncertainty of the certified value (U_{CRM}) with a coverage factor k given as:

$$U_{\text{CRM}} = k \cdot \sqrt{u_{\text{char}}^2 + u_{\text{hom}}^2 + u_{\text{trn}}^2 + u_{\text{sts}}^2} \quad \text{Equation 5}$$

7.1 Uranium isotope amount ratios

The certified values of the uranium isotope amount ratios for IRMM-2329P were derived from the TIMS/MTE measurements in the IRMM-2329 solution, with confirmation by the TIMS/MTE and MC-ICPMS verification measurements at IAEA-SGAS. The uranium isotope amount ratios in IRMM-2329P are given in mol·mol⁻¹.

From the certified values for the uranium isotope amount ratio, the isotope amount fractions can be derived by dividing the isotope amount ratios for the various isotopes by the sum of the isotope amount ratios:

$$n(^x\text{U})/n(\text{U}) = \frac{n(^x\text{U})/n(^{238}\text{U})}{\sum_{i=\{234;235;236;238\}} n(^i\text{U})/n(^{238}\text{U})} \quad \text{Equation 6}$$

Moreover, the molar mass of the uranium can be calculated by multiplication of the isotope amount fractions with the molar mass of the respective isotope:

$$M(\text{U}) = \sum_{i=\{234;235;236;238\}} n(^i\text{U})/n(\text{U}) \cdot M(^i\text{U}) \quad \text{Equation 7}$$

For the calculation of the uranium molar mass, the molar mass of the individual isotopes of uranium (²³⁴U, ²³⁵U, ²³⁶U and ²³⁸U) have been taken from the most recent atomic mass evaluation (AME2016, [26]).

Finally, the isotope mass fractions for the different uranium isotopes can be calculated by multiplication of the isotope amount fraction with the isotope molar mass, followed by division by the molar mass of the uranium:

$$m(^x\text{U})/m(\text{U}) = \frac{n(^x\text{U})/n(\text{U}) \cdot M(^x\text{U})}{M(\text{U})} \quad \text{Equation 8}$$

The certified and calculated derived values related to the uranium isotopic composition for IRMM-2329P are summarised in Table and in Annex 12.

Table 4: Certified and derived values and their uncertainties related to the uranium isotopic composition of IRMM-2329P.

	Certified values	$U_{\text{CRM}} (^1)$	$U_{\text{CRM,rel}} (^1)$
$n(^{234}\text{U})/n(^{238}\text{U})$ [mol·mol ⁻¹]	0.00034083	0.00000019	0.056 %
$n(^{235}\text{U})/n(^{238}\text{U})$ [mol·mol ⁻¹]	0.033902	0.000012	0.036 %
$n(^{236}\text{U})/n(^{238}\text{U})$ [mol·mol ⁻¹]	0.00003021	0.00000012	0.40 %
$n(^{234}\text{U})/n(\text{U})$ [mol·mol ⁻¹]	0.00032953	0.00000018	0.055 %
$n(^{235}\text{U})/n(\text{U})$ [mol·mol ⁻¹]	0.032778	0.000011	0.034 %

$n(^{236}\text{U})/n(\text{U})$ [mol·mol ⁻¹]	0.00002921	0.00000012	0.41 %
$n(^{238}\text{U})/n(\text{U})$ [mol·mol ⁻¹]	0.966863	0.000011	0.0017 %
$M(\text{U})$ [g·mol ⁻¹]	237.950848	0.000034	0.000021 %
$m(^{234}\text{U})/m(\text{U})$ [g·g ⁻¹]	0.00032412	0.00000018	0.056 %
$m(^{235}\text{U})/m(\text{U})$ [g·g ⁻¹]	0.032378	0.000011	0.049 %
$m(^{236}\text{U})/m(\text{U})$ [g·g ⁻¹]	0.00002897	0.00000012	0.41 %
$m(^{238}\text{U})/m(\text{U})$ [g·g ⁻¹]	0.967269	0.000011	0.0017 %

⁽¹⁾ Expanded ($k = 2$) and rounded uncertainty.

7.2 Uranium amount per particle

The certified value of the uranium amount per particle in IRMM-2329P was determined based on isotope dilution mass spectrometry (IDMS) measurements and is given in fmol. A full uncertainty budget was established in accordance with the "Guide to the Expression of Uncertainty in Measurement" [3] and can be found in Annex 9.

The uncertainty related to degradation during long-term storage was not yet determined and therefore not considered for the current certification. In case the regular postmonitoring program would reveal unexpected significant changes of the uranium amount per particle over time, an uncertainty for the long-term stability has to be taken into account when the validity period of the certificate is extended.

Because of the sufficient numbers of the degrees of freedom of the combined uncertainty, a coverage factor $k = 2$ was applied, which corresponds to a confidence interval of 95 %. The various uncertainty contributions together with the certified value and expanded relative uncertainty $U_{\text{CRM,rel}}$ are summarised in Table 4.

Table 5: Certified values and their uncertainties for the uranium amount (in fmol) per particle of IRMM-2329P. The quantity $U_{\text{hom,rel}}$ is calculated by combining $U_{\text{swb,rel}}$ and $U_{\text{sbb,rel}}$.

	Certified value	$U_{\text{char,rel}}$	$U_{\text{hom,rel}}$	$U_{\text{CRM,rel}}^{(1)}$
IRMM-2329P	15.1 fmol	4.0 %	7.8 %	17 %

⁽¹⁾ Expanded ($k = 2$) and rounded uncertainty.

From the certified value of the uranium amount per particle, the uranium mass per particle can be derived by multiplication of the certified value by the molar mass of the uranium, $M = 237.950848(34)$ g·mol⁻¹, as described in section 7.1 and Table .

Furthermore, the number of uranium atoms per particle can be derived by dividing the uranium amount per particle by Avogadro's constant⁴, $N_{\text{A}} = 6.02214086(14) \cdot 10^{23}$ mol⁻¹.

Table lists all the certified values and their expanded uncertainties, all given with a coverage factor k of 2.

⁴ The NIST Reference on Constants, Units, and Uncertainty (accessed 22/02/2018). <https://physics.nist.gov/cgi-bin/cuu/Value?na>

Table 6: Certified values and their uncertainties related to the uranium amount per particle of IRMM-2329P.

Property value	Certified value	$U_{\text{CRM}}^{(1)}$	$U_{\text{CRM,rel}}^{(1)}$
Uranium amount per particle	15.1 fmol	2.6 fmol	17 %
Uranium mass per particle	3.58 pg	0.63 pg	17 %
Number of uranium atoms per particle	$9.1 \cdot 10^9$	$1.6 \cdot 10^9$	17 %

⁽¹⁾ Expanded ($k = 2$) and rounded uncertainty.

8 Metrological traceability and commutability

8.1 Metrological traceability

Traceability of the obtained results is based on the traceability of all relevant input factors. Instruments were verified and calibrated with tools ensuring traceability to the International System of units (SI).

The certified values for the uranium isotope amount ratios of IRMM-2329P are traceable to the International System of Units (SI) via the certified values of the certificate of the IRMM-074.

The certified values for the uranium amount per particle for IRMM-2329P are traceable to the SI via the certified values of the certificate of the IRMM-058.

8.2 Commutability

Many measurement procedures include one or more steps which select specific analytes from the sample for the subsequent whole measurement process. Often the complete identity of these 'intermediate analytes' is not fully known or taken into account. Therefore, it is difficult to mimic all analytically relevant properties of real samples within a CRM. The degree of equivalence in the analytical behaviour of real samples and a CRM with respect to various measurement procedures is summarised in a concept called 'commutability of a reference material'. There are various definitions that define this concept. For instance, the CLSI Guideline C53-A [28] recommends the use of the following definition for the term commutability:

"The equivalence of the mathematical relationships among the results of different measurement procedures for an RM and for representative samples of the type intended to be measured."

The commutability of a CRM defines its fitness for use and is therefore a crucial characteristic when applying different measurement methods. When the commutability of a CRM is not established, the results from routinely used methods cannot be legitimately compared with the certified value to determine whether a bias does not exist in calibration, nor can the CRM be used as a calibrant.

The IRMM-2329P uranium particles were produced from synthetic micro particles produced via a spray-pyrolysis procedure employing a thermal treatment to improve stability. The analytical behaviour will be the same as for typical uranium micro particles.

This reference material is tailor-made to be used by the nuclear safeguards community as calibrant, QC sample and reference material mainly for mass spectrometry analysis.

9 Instructions for use

9.1 Safety information

The IRMM-2329P contains low-level radioactive material in exempted quantities. The planchet should be handled with great care and by experienced personnel in a laboratory suitably equipped for the safe handling of low-level radioactive materials.

9.2 Storage conditions

The material should be stored in a dry environment at room temperature. Some silica bags should be placed with the material to optimise its storage. Care should be taken to identify the particle bearing surface and to prevent any cross-contamination onto the sample surface.

Please note that the European Commission cannot be held responsible for changes that happen during storage of the material at the customer's premises, especially for opened units.

9.3 Preparation and use of the material

However, it is recommended to pay attention as to the correct side on which the particles are deposited when manipulating the planchet and placing it back into its case.

9.4 Minimum sample intake

The minimal sample intake with regard to the isotope amount ratios of IRMM-2329P is 1 particle.

The minimal sample intake for the uranium amount per particle in IRMM-2329P is 10 particles, unless the analytical method is capable of discriminating between particles which belong to the major particle population and particles containing double the amount of uranium per particle compared to the major particle population, in which case the minimal sample intake is 1 particle.

9.5 Use of the certified values

The main purpose of these materials is to assess method performance, i.e. for checking accuracy of analytical results and calibration. As any reference material, they can be used for establishing control charts or validation studies.

The use of IRMM-2329P as calibrant for isotope ratio measurements using mass spectrometric techniques such as LG-SIMS, FT-TIMS, TIMS with micro-manipulation and LA-MC-ICPMS, is restricted to small surface areas below a square of $20\mu\text{m} \times 20\mu\text{m}$ around the particles.

9.6 Use as a calibrant

The uncertainty of the certified value shall be taken into account in the estimation of the measurement uncertainty.

Comparing an analytical result with the certified value

A result is unbiased if the combined standard uncertainty of measurement and certified value covers the difference between the certified value and the measurement result (see also ERM Application Note 1, www.erm-crm.org).

When assessing the method performance, the measured values of the CRMs are compared with the certified values. The procedure is summarised here:

1. Calculate the absolute difference between mean measured value and the certified value (Δ_{meas}).
2. Combine the measurement uncertainty (u_{meas}) with the uncertainty of the certified value (u_{CRM}):

$$u_{\Delta} = \sqrt{u_{\text{meas}}^2 + u_{\text{CRM}}^2}.$$

3. Calculate the expanded uncertainty (U_{Δ}) from the combined uncertainty (u_{Δ}) using an appropriate coverage factor, corresponding to a level of confidence of approximately 95 %.
4. If $\Delta_{\text{meas}} \leq U_{\Delta}$ then no significant difference exists between the measurement result and the certified value, at a confidence level of approximately 95 %.

9.7 Use in quality control charts

The materials can be used for quality control charts. Using CRMs for quality control charts has the added value that a trueness assessment is built into the chart.

References

- [1] ISO 17034:2016 General requirements for the competence of reference material producers.
- [2] ISO Guide 35:2006 Reference materials – General and statistical principles for certification.
- [3] ISO/IEC Guide 98-3, Guide to the Expression of Uncertainty in Measurement (GUM, 1995), International Organization for Standardization, Geneva, Switzerland, 2008
- [4] International Atomic Energy Agency. Treaty on the non-proliferation of nuclear weapons, 22 April 1970.
- [5] International Atomic Energy Agency. Model Protocol Additional to the Agreement(s) between State(s) and the International Atomic Energy Agency for the Application of Safeguards, September 1997 COUNCIL
- [6] COUNCIL REGULATION (EURATOM) No 1314/2013 on the Research and Training Programme of the European Atomic Energy Community
- [7] International Atomic Energy Agency. The IAEA Safeguards Analytical Laboratories, October 2012.
- [8] L. Sangély, J. Poths, H. Siegmund, T. Tanpraphan, O. Bildstein, M. Aleshin, and A. Schwanhäusser. Implementation of Large-Geometry SIMS for Safeguards: 4 years later. In *ESARDA 37th Annual Meeting Proceedings*, 66:873–883 (2015).
- [9] NUSIMEP-9: Uranium isotope amount ratios and uranium mass in uranium micro-particles, C. Venchiarutti, S. Richter, R. Middendorp, Y. Aregbe, EUR 29822 EN, ISBN 978-92-76-09182-0; doi:10.2760/533028
- [10] S. Richter, H. Kühn, Y. Aregbe, M. Hedberg, J. Horta-Domenech, K. Mayer, E. Zuleger, S. Bürger, S. Boulyga, A. Köpf, J. Poths, K. Matthew. Improvements for Routine Uranium Isotope Ratio Measurements using the Modified Total Evaporation Method, *Journal of Analytical Atomic Spectrometry*, 26:550 (2011).
- [11] ASTM Standard Document C1832, available through the ASTM website: <https://www.astm.org/Standards/C1832.htm>
- [12] R. Middendorp, M. Dürr, A. Knott, F. Pointurier, D. Ferreira Sanchez, V. Samson, and D. Grolimund. Characterization of the Aerosol-Based Synthesis of Uranium Particles as a Potential Reference Material for Microanalytical Methods. *Analytical Chemistry*, 89(8):4721–4728 (2017). doi:10.1021/acs.analchem.7b00631.
- [13] R. Middendorp. Synthesis and Stability Assessment of Uranium Micro Particles: Providing Reference Materials for Nuclear Safeguards. PhD thesis, Forschungszentrum Jülich GmbH (2018).
- [14] S. Richter, C. Hennessy, U. Jacobsson, R. Bujak, C. Venchiarutti, J. Truyens, Y. Aregbe, "CERTIFICATION REPORT, Preparation and Certification of the Uranium Nitrate Solution Series IRMM-2019-2029, Revision of the IRMM-019-029 Series of UF₆ Materials", EUR 29299 EN
- [15] S. Richter, C. Venchiarutti, C. Hennessy, U. Jacobsson, R. Bujak, J. Truyens, Y. Aregbe, "Preparation and certification of the uranium nitrate solution reference materials series IRMM-2019 to IRMM-2029 for the isotopic composition", published in the *Journal of Radioanalytical and Nuclear Chemistry*, 318:1359–1368 (2018)
- [16] R. Middendorp, M. Dürr, I. Niemeyer, and D. Bosbach. Micro Particle Suspensions for Preparation of Reference Materials for Particle Analysis Methods in Safeguards. *ESARDA Bulletin*, 54:23–30 (2017).
- [17] R. Middendorp, M. Klinkenberg, M. Dürr, Uranium oxide microparticle suspensions for the production of reference materials for micro-analytical methods, *J Radioanal Nucl Chem* **318**, 907–914 (2018).
- [18] S. Sakurai and S. Tachimori. Density Equation of Aqueous Solution Containing Plutonium(IV), Uranium(VI) and Nitric Acid. *Journal of Nuclear Science and Technology*, 33(2):187–189 (1996).
- [19] T. Linsinger, J. Pauwels, A. van der Veen, H. Schimmel and A. Lamberty. Homogeneity and stability of reference materials. *Accreditation and Quality Assurance*, 6:20–25 (2001).

- [20] T. Linsinger, J. Pauwels, A. Lamberty, H. Schimmel, A. van der Veen and L. Siekmann. Estimating the uncertainty of stability for matrix CRMs. *Fresenius Journal of Analytical Chemistry*, 370:183-188 (2001).
- [21] S. Mialle, S. Richter, J. Truyens, C. Hennessy, U. Jacobsson and Y. Aregbe. Certification of the uranium hexafluoride (UF₆) isotopic composition: The IRMM-019 to IRMM-029 series (2014).
- [22] I. Grenthe, I. Buck, E. Drozdynski, J. Fujino, T. Albrecht-Schmitt, and S. Wolf (editors). Uranium, chapter 5, p.253-698. *The Chemistry of the Actinide and Transactinide Elements*. Springer, 3rd edition (2006).
- [23] Y. Aregbe, J. Truyens, R. Kips, S. Richter, E. Stefaniak, H. Kühn, and M. Kraiem. *NUSIMEP-6: Uranium isotope amount ratios in uranium particles* (2008).
- [24] J. Truyens, E. Stefaniak, S. Mialle, and Y. Aregbe. *NUSIMEP-7: Uranium isotope amount ratios in uranium particles* (2011).
- [25] M. Milton and T. Quinn. Primary methods for the measurement of amount of substance. *Metrologia*, 38:289-296 (2001).
- [26] ISO/IEC Guide 99:2007 International vocabulary of metrology -- Basic and general concepts and associated terms (VIM).
- [27] M. Wand, G. Audi, F. Kondev, W. Huang, S. Naimi and X. Xu. The AME2016 atomic mass evaluation (II). Tables, graphs and references. *Chinese Physics C*, 41(3): 030003 (2016).
- [28] H. Vesper, H. Emons, M. Gnezda, C. Jain, W. Miller, R. Rej, G. Schumann, J. Tate, L. Thienpont and J. Vaks. *Characterization and Qualification of Commutable Reference Materials for Laboratory Medicine; Approved Guideline*, CLSI document C53-A (2010).

List of abbreviations and definitions

ANOVA	Analysis of variance
AP	Additional protocol
CRM	Certified reference material
EC	European Commission
ES	Environmental sampling
FZJ	Forschungszentrum Jülich
IAEA	International Atomic Energy Agency
ID	Isotope dilution
IDMS	Isotope dilution mass spectrometry
JRC	Joint Research Centre
k	Coverage factor
LG-SIMS	Large-geometry secondary ion mass spectrometry
m	Mass of substance; Number of sets in simulation
M	Molar mass
MC-ICPMS	Multi-collector inductively coupled plasma mass spectrometry
MS_{between}	Mean of squares between-unit from an ANOVA
MS_{within}	Mean of squares within-units from an ANOVA
MTE	Modified total evaporation
n	Amount of substance/number or replicates
N	Number of particles
N_A	Avogadro's constant
<i>NML</i>	<i>Nuclear Material Laboratory</i>
NUSIMEP	Nuclear Signatures Inter-laboratory Measurement Evaluation Programme
NWAL	Network of analytical laboratories
OPS	Optical particle sizer
p	Probability
R	Isotope amount ratio
RM	Reference material
SGAS	Safeguards Analytical Services
s_{bb}	Between-bottle inhomogeneity standard deviation
SEM	Scanning electron microscope
SI	International system of units
SIMS	Secondary ion mass spectrometry
s_{wb}	Within-unit standard deviation
t	Time
TIMS	Thermal ionisation mass spectrometry
U	Expanded uncertainty

U	Standard uncertainty
U_{bb}^*	Standard uncertainty related to a maximum between-unit inhomogeneity that could be hidden by method intermediate precision
U_{bb}	Standard uncertainty related to a possible between-unit inhomogeneity
U_{char}	Standard uncertainty due to material characterisation
U_{CRM}	Combined standard uncertainty of the certified value
$U_{CRM,max}$	Maximum permissible standard uncertainty of the certified value at which the possible within-unit inhomogeneity would be negligible
U_{CRM}	Expanded uncertainty of the certified value
U_{hom}	Standard uncertainty of the homogeneity study
U_{lts}	Standard uncertainty of the long-term stability
U_{meas}	Standard measurement uncertainty
U_{tm}	Standard uncertainty due to short-term stability (transport)
$U_{wb,max}$	Maximum permissible uncertainty due to within-unit inhomogeneity
VIM	International vocabulary on metrology
VOAG	Vibrating orifice aerosol generator
ν	Degrees of freedom
X_{CRM}	Certified value

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Annex 1. Certificate of IRMM-023



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**CERTIFIED REFERENCE MATERIAL
IRMM-023
CERTIFICATE OF ANALYSIS**

URANIUM HEXAFLUORIDE		
	Isotope amount ratio	
	Certified value ¹⁾ [mol/mol]	Uncertainty ²⁾ [mol/mol]
$n(^{234}\text{U})/n(^{238}\text{U})$	0.00033950	0.00000011
$n(^{235}\text{U})/n(^{238}\text{U})$	0.0338814	0.0000054
$n(^{236}\text{U})/n(^{238}\text{U})$	0.0000001153	0.0000000017
	Isotope amount fraction (x 100)	
	Certified value ³⁾ [mol/mol]	Uncertainty ²⁾ [mol/mol]
$n(^{234}\text{U})/n(\text{U})$	0.032827	0.000010
$n(^{235}\text{U})/n(\text{U})$	3.27603	0.00051
$n(^{236}\text{U})/n(\text{U})$	0.00001115	0.00000016
$n(^{238}\text{U})/n(\text{U})$	96.69113	0.00051
	Isotope mass fraction (x 100)	
	Certified value ^{3) 4)} [g/g]	Uncertainty ²⁾ [g/g]
$m(^{234}\text{U})/m(\text{U})$	0.032288	0.000010
$m(^{235}\text{U})/m(\text{U})$	3.23601	0.00050
$m(^{236}\text{U})/m(\text{U})$	0.00001106	0.00000016
$m(^{238}\text{U})/m(\text{U})$	96.73169	0.00050
M(U)	Molar mass	
	Certified value ^{3) 4)} [g/mol]	Uncertainty ²⁾ [g/mol]
	237.950965	0.000016

All following pages are an integral part of the certificate.
Page 1 of 3

- 1) The certified values are traceable to the International System of units (SI) via IRMM-3636a.
- 2) The 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.
- 3) These values are calculated using the isotope amount ratios and therefore traceable to the SI. The calculation of $n(\text{U})$, $m(\text{U})$ and $M(\text{U})$ includes the contributions from the isotopes ^{234}U , ^{235}U , ^{236}U and ^{238}U .
- 4) These values are calculated using the values listed below from Wang et al., *The AME 2016 atomic mass evaluation, Chinese Physics C Vol. 41, No. 3 (2017) 030003*:
- $M(^{234}\text{U}) = 234.0409504 \pm 0.0000024 \text{ g}\cdot\text{mol}^{-1} (k = 2)$
 $M(^{235}\text{U}) = 235.0439282 \pm 0.0000024 \text{ g}\cdot\text{mol}^{-1} (k = 2)$
 $M(^{236}\text{U}) = 236.0455662 \pm 0.0000024 \text{ g}\cdot\text{mol}^{-1} (k = 2)$
 $M(^{238}\text{U}) = 238.0507870 \pm 0.0000032 \text{ g}\cdot\text{mol}^{-1} (k = 2)$

This certificate is valid for 10 years.

Former date of the certification: 06/06/2014.
Geel, revised

27 AUG. 2014

Signed:



Dr. Arjan Plompen
European Commission
Joint Research Centre
Directorate G – Nuclear Safety and Security
G.2 - Standards for Nuclear Safety, Security and
Safeguards Unit
Reijeseweg 111
B-2440 Geel, Belgium

DESCRIPTION OF THE MATERIAL

IRMM-023 is a uranium hexafluoride (UF_6) Isotopic Reference Material as certified above. It is supplied in monel ampoules.

ANALYTICAL METHODS USED FOR CERTIFICATION

The uranium hexafluoride gas was hydrolysed and converted into uranyl nitrate solution. The certified values for this solution were established by Thermal Ionisation Mass Spectrometry (TIMS) using the Modified Total Evaporation and Double Spike techniques (according to ASTM C1832 and ASTM C1871, respectively). Confirmation measurements were performed on uranium hexafluoride in gaseous form using Gas Source Mass Spectrometry (GSMS).

SAFETY INFORMATION

The IRMM-023 contains radioactive material. The ampoule should be handled with great care and by experienced personnel in a laboratory suitably equipped for the safe handling of radioactive materials.

INSTRUCTIONS FOR USE AND INTENDED USE

This reference material is intended to be used as a calibrant for isotope mass spectrometry measurements. Prior to use, it is recommended to cool the ampoule with a dry ice-ethanol mixture at about $-70\text{ }^{\circ}\text{C}$ and evacuate the ampoule under vacuum (pressure below 1×10^{-2} mbar). For safety reasons, the vacuum line should be connected to a cold trap.

STORAGE

The ampoule should be stored at room temperature.

However, the European Commission cannot be held responsible for changes that happen during storage of the material at the customer's premises, especially of opened samples.

LEGAL NOTICE

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NOTE

The certification report for the IRMM-019 to IRMM-029 series is available on the internet (<https://crm.jrc.ec.europa.eu>). A paper copy can be obtained from the Joint Research Centre on request.

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Annex 2. Certificate of IRMM-029



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**CERTIFIED REFERENCE MATERIAL
IRMM-029
CERTIFICATE OF ANALYSIS**

URANIUM HEXAFLUORIDE		
	Isotope amount ratio	
	Certified value ¹⁾ [mol/mol]	Uncertainty ²⁾ [mol/mol]
$n(^{234}\text{U})/n(^{238}\text{U})$	0.00084444	0.00000037
$n(^{235}\text{U})/n(^{238}\text{U})$	0.044052	0.000013
$n(^{236}\text{U})/n(^{238}\text{U})$	0.0105563	0.0000022
	Isotope amount fraction (x 100)	
	Certified value ³⁾ [mol/mol]	Uncertainty ²⁾ [mol/mol]
$n(^{234}\text{U})/n(\text{U})$	0.080007	0.000035
$n(^{235}\text{U})/n(\text{U})$	4.1737	0.0012
$n(^{236}\text{U})/n(\text{U})$	1.00017	0.00021
$n(^{238}\text{U})/n(\text{U})$	94.7461	0.0012
	Isotope mass fraction (x 100)	
	Certified value ^{3) 4)} [g/g]	Uncertainty ²⁾ [g/g]
$m(^{234}\text{U})/m(\text{U})$	0.078709	0.000034
$m(^{235}\text{U})/m(\text{U})$	4.1236	0.0012
$m(^{236}\text{U})/m(\text{U})$	0.99237	0.00021
$m(^{238}\text{U})/m(\text{U})$	94.8054	0.0012
	Molar mass	
	Certified value ^{3) 4)} [g/mol]	Uncertainty ²⁾ [g/mol]
M(U)	237.902026	0.000036

All following pages are an integral part of the certificate.
Page 1 of 3

- 1) The certified values are traceable to the International System of units (SI) via IRMM-074/10.
- 2) The 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.
- 3) These values are calculated using the isotope amount ratios and therefore traceable to the SI. The calculation of $n(U)$, $m(U)$ and $M(U)$ includes the contributions from the isotopes ^{234}U , ^{235}U , ^{236}U and ^{238}U .
- 4) These values are calculated using the values listed below from Wang et al., *The AME 2016 atomic mass evaluation*, *Chinese Physics C Vol. 41, No. 3 (2017) 030003*:
- $M(^{234}\text{U}) = 234.0409504 \pm 0.0000024 \text{ g}\cdot\text{mol}^{-1} (k = 2)$
 $M(^{235}\text{U}) = 235.0439282 \pm 0.0000024 \text{ g}\cdot\text{mol}^{-1} (k = 2)$
 $M(^{236}\text{U}) = 236.0455662 \pm 0.0000024 \text{ g}\cdot\text{mol}^{-1} (k = 2)$
 $M(^{238}\text{U}) = 238.0507870 \pm 0.0000032 \text{ g}\cdot\text{mol}^{-1} (k = 2)$

This certificate is valid for 10 years.

Former date of the certification: 06/08/2014.
Geel, revised

Signed:



Dr. Arjan Plompen
European Commission
Joint Research Centre
Directorate G – Nuclear Safety and Security
G.2 - Standards for Nuclear Safety, Security and
Safeguards Unit
Retieseweg 111
B-2440 Geel, Belgium

27 AUG. 2018

DESCRIPTION OF THE MATERIAL

IRMM-029 is a uranium hexafluoride (UF₆) Isotopic Reference Material as certified above. It is supplied in monel ampoules.

ANALYTICAL METHODS USED FOR CERTIFICATION

The uranium hexafluoride gas was hydrolysed and converted into uranyl nitrate solution. The certified values for this solution were established by Thermal Ionisation Mass Spectrometry (TIMS) using the Modified Total Evaporation technique (according to ASTM C1832). Confirmation measurements were performed on uranium hexafluoride in gaseous form using Gas Source Mass Spectrometry (GSMS).

SAFETY INFORMATION

The IRMM-029 contains radioactive material. The ampoule should be handled with great care and by experienced personnel in a laboratory suitably equipped for the safe handling of radioactive materials.

INSTRUCTIONS FOR USE AND INTENDED USE

This reference material is intended to be used as a calibrant for isotope mass spectrometry measurements. Prior to use, it is recommended to cool the ampoule with a dry ice-ethanol mixture at about -70 °C and evacuate the ampoule under vacuum (pressure below 1×10⁻² mbar). For safety reasons, the vacuum line should be connected to a cold trap.

STORAGE

The ampoule should be stored at room temperature. However, the European Commission cannot be held responsible for changes that happen during storage of the material at the customer's premises, especially of opened samples.

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NOTE

The certification report for the IRMM-019 to IRMM-029 series is available on the internet (<https://crm.jrc.ec.europa.eu>). A paper copy can be obtained from the Joint Research Centre on request.

European Commission – Joint Research Centre
Directorate G – Nuclear Safety and Security
G.2 - Standards for Nuclear Safety, Security and Safeguards Unit
Retieseweg 111, B - 2440 Geel (Belgium)

Annex 3. Measurement Certificate for IRMM-2329



EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Directorate G - Nuclear Safety and Security
Unit G.2 - Standards for Nuclear Safety, Security and Safeguards (SN3S)

Certificate of Reference Measurement #3867

Customer

Company/Institute	JRC
Division	Unit G.2
Address	Rotieseweg 11
	2440 Geel
	Belgium
Requestor	JRC-G.2

Sample Information

Sample type	Uranium nitrate solution for particle production
Sample reference	IRMM-2329
Date of receipt of sample(s)	04/04/2017
JRC sample identification	27004
Condition of sample	Uranium nitrate solution, 2mg U / mL

Measurement Results

Analyte	Result	Unit	Method
$n(^{234}\text{U})/n(^{238}\text{U})$	0.00034083(19)	mol / mol	TIMS/MTE
$n(^{235}\text{U})/n(^{238}\text{U})$	0.033902(12)	mol / mol	TIMS/MTE
$n(^{236}\text{U})/n(^{238}\text{U})$	0.00003021(12)	mol / mol	TIMS/MTE

Derived Quantities

Molar Mass	237.950844(34)		
Amount fraction (x100)		Mass fraction (x100)	
$n(^{234}\text{U})/n(\text{U})$	0.032953(18)	$m(^{234}\text{U})/m(\text{U})$	0.032412(18)
$n(^{235}\text{U})/n(\text{U})$	3.2778(11)	$m(^{235}\text{U})/m(\text{U})$	3.2378(11)
$n(^{236}\text{U})/n(\text{U})$	0.002921(12)	$m(^{236}\text{U})/m(\text{U})$	0.002897(12)
$n(^{238}\text{U})/n(\text{U})$	96.6863(11)	$m(^{238}\text{U})/m(\text{U})$	96.7269(11)

This report may only be reproduced in full and with the written consent of the requestor.
Results relate only to samples analysed. No feedback within 4 weeks constitutes acceptance of the report.
Potential sample rests may be destroyed after this period.

Uncertainties:

All uncertainties indicated are expanded uncertainties $U = k \cdot u_c$ where u_c is the combined standard uncertainty calculated according to the ISO/BIPM guide¹. They are given in parentheses and include a coverage factor $k=2$. They apply to the last two digits of the value. The values certified are traceable to the SI. The traceability to SI is established through IRMM-074. Uncertainties include an extrapolated contribution for homogeneity, as described in².

Analytical measurement procedure:

- Sample preparation has been accomplished by J. Truyens
- Mass spectrometric measurements have been accomplished by S. Richter
- Analytical method/technique used : TIMS/MTE
- The atomic masses, used in the calculations, are³:
- The half-lives used in the calculations are: N/A
- The mass spectrometer was calibrated using IRMM-074
- Quality control samples used were IRMM-075/1 and IRMM-075/2

Date of analysis (dd/mm/yyyy)	27/04/2017
Date of internal analysis report (dd/mm/yyyy)	14/06/2017
Certification date normalised to (reference date)	27/04/2017 at 12:00 h

Backup Files and Raw Data

Folder:
"G:\JRC.G.2\Nuclear Safeguards\Nuclear\Particles\Certification U Particle RM IRMM-2329-2330\TIMS MTE Measurements of Base Solutions IRMM-2329-2330"

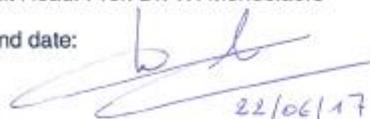
Data Files:
"T170424 MTE IRMM-2329-SEM-2330-SEM-FAR.xls"
"Calculation homogeneity extrapolation-2 - applied for IRMM-2329-2330.xls"
"IRMM-2329.SMU"

Annexes

Copy of Internal Analysis Report (2 pages)

JRC G.2 Unit Head: Prof. Dr. W. Mondelaers

Signature and date:



22/06/17

References:

- 1 International Organisation for Standardisation, Guide to the Expression of Uncertainty in Measurements, ©ISO, ISBN 92-87-10188-9, Geneva, Switzerland 1993
- 2 Mialle, S., Richter, S., Hennessy, C., Truyens, J., Jacobson, U., et al. "Certification of uranium hexafluoride reference materials for isotopic composition", 8th International Conference on Isotopes, Chicago, Aug. 24-29, 2014. J Radioanal Nucl Chem, Vol 305, 2015, pp. 255-266.
- 3 G. Audi and A.H. Wapstra, The 2003 atomic mass evaluation, Nuclear Physics A 729(2003) 337-676

Retieseweg 111, B-2440 Geel - Belgium, Telephone: (32-14) 571 211, <http://irmm.jrc.ec.europa.eu>

Annex 4. Certificate of IRMM-074 series

CERTIFICATE

ISOTOPIC REFERENCE MATERIAL IRMM-074

The Isotopic Reference Material IRMM-074 is a set of mixtures of uranium isotopes ^{233}U , ^{235}U and ^{238}U with molar ratios certified as follows:

Code Number	Molar Isotope Abundance Ratio		
	$n(^{233}\text{U})/n(^{235}\text{U})$ $U = 0.025\%$ (relative)	$n(^{233}\text{U})/n(^{238}\text{U})$ $U = 0.025\%$ (relative)	$n(^{235}\text{U})/n(^{238}\text{U})$ $U = 0.015\%$ (relative)
IRMM-074/1	1.026 85	1.027 11	1.000 254
IRMM-074/2	0.307 993	0.308 072	1.000 258
IRMM-074/3	0.010 228 8	0.010 231 4	1.000 259
IRMM-074/4	0.003 073 58	0.003 074 37	1.000 259
IRMM-074/5	0.001 030 61	0.001 030 88	1.000 259
IRMM-074/6	0.000 307 778	0.000 307 858	1.000 259
IRMM-074/7	0.000 102 603	0.000 102 629	1.000 259
IRMM-074/8	0.000 030 801 1	0.000 030 809 1	1.000 259
IRMM-074/9	0.000 008 158 7	0.000 008 160 8	1.000 259
IRMM-074/10	0.000 001 018 86	0.000 001 019 13	1.000 259

The Isotopic Reference Material is intended for the verification and correction of non-linearities of the entire mass spectrometer measurement system.

NOTES

1. This Isotopic Reference Material is traceable to the international SI unit for amount of substance - the mole - via synthetic mixtures prepared at IRMM. Measurements calibrated against these Isotopic Reference Materials will, therefore, also be traceable to the SI unit system.

07/2006

2. The uncertainties as specified in the table can be considered as expanded uncertainties U where $k = 2$. The value of the standard uncertainty can therefore be derived: $u_c = U \cdot 0.5$
The uncertainties given are supported by calculation of the combined uncertainty following the ISO/GUM recommendations¹ and are based on measured values of the isotopic enrichments, the weights of oxides and solutions, and of the impurity levels. The uncertainties were also confirmed through comparison measurements made on samples of IRMM-072, IRMM-199 and CRM-U-500 (DOE/NBL).
3. Values for molar isotope abundance ratios are valid for June 2005.
4. The Isotopic Reference Material IRMM-074 consists of a set of 10 units. Each unit consists of approximately 0.2 mg uranium as uranyl nitrate in 2 mL 1 M nitric acid solution in a sealed quartz glass ampoule.
5. The atomic masses, used in the calculations, are²
 - ^{233}U : 233.039 627 0(60) g·mol⁻¹
 - ^{234}U : 234.040 944 7(44) g·mol⁻¹
 - ^{235}U : 235.043 922 2(42) g·mol⁻¹
 - ^{236}U : 236.045 561 0(42) g·mol⁻¹
 - ^{238}U : 238.050 783 5(44) g·mol⁻¹

6. The vial should be opened with great care and by experienced personnel in a laboratory environment suitably equipped for the safehandling of radioactive materials.
7. Full details on the certification procedure can be found in the Certification Report EUR 22270 EN³.

Chemical purification of the $^{233}\text{U}_3\text{O}_8$, $^{235}\text{U}_3\text{O}_8$ and $^{238}\text{U}_3\text{O}_8$ starting materials was performed by R Eykens.

Weighing and preparation of the Isotopic Reference Material was performed by F Hendrickx and R Eykens. Characterization of the enriched isotopes from which the set was prepared and verification measurements on the mixtures, were performed by S Richter on samples prepared by F Kehoe and A Alonso Muñoz. The ampoulation of this Isotopic Reference Material was accomplished by G Van Baelen and A Verbruggen.

The overall coordination leading to the establishment, certification and issuance of this Isotopic Reference Material set and of the preparation and issuance of the certificate was performed by A Verbruggen.

B-2440 GEEL
July 2006

P Taylor
Head
Isotope Measurements Unit

R Wellum
IRMM Safeguards Coordinator

¹ International Organisation for Standardisation, Guide to the expression of Uncertainty in Measurement, ©ISO, ISBN 92-87-10188-9, Geneva, Switzerland, 1993

² G. Audi and A.H. Wapstra, The 1993 atomic mass evaluation, Nucl Phys A565 (1993) 1-85.

³ A. Verbruggen, A. Alonso, R. Eykens, F. Hendrickx, F. Kehoe, H. Kühn, S. Richter, G. Van Baelen, R. Wellum, Preparation and certification of IRMM-074, a new set of uranium isotope mixtures for calibration of mass spectrometers, Report EUR 22270 EN

Annex 5. Certificate of IRMM-184



EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Directorate G – Nuclear Safety and Security
G.2 – Standards for Nuclear Safety, Security and Safeguards Unit

CERTIFIED REFERENCE MATERIAL
IRMM – 184

CERTIFICATE OF ANALYSIS

Uranium in nitric acid solution		
	Isotope amount ratios	
	Certified value ¹⁾ [mol/mol]	Uncertainty ²⁾ [mol/mol]
$n(^{233}\text{U})/n(^{238}\text{U})$	< 0.000000002	/
$n(^{234}\text{U})/n(^{238}\text{U})$	0.000053138	0.000000032
$n(^{235}\text{U})/n(^{238}\text{U})$	0.0072623	0.0000022
$n(^{236}\text{U})/n(^{238}\text{U})$	0.00000012446	0.00000000053

¹⁾ The certified values are traceable to the International System of units (SI).

²⁾ The 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.

There is no minimum sample intake to be taken into account.

The certificate is valid for 3 years; the validity may be extended after further tests on the stability of the material are carried out.

The certificate is a revision of the original certificate of 1987, which was revised in 1993, 1999 and 2005.

Geel, September 2019

Signed: _____

18 SEP. 2019

Dr. Arjan Plompen
European Commission
Joint Research Centre
Directorate G – Nuclear Safety and Security
G.2 – Standards for Nuclear Safety, Security and Safeguards Unit
Retieseweg 111
B-2440 Geel, Belgium

The following values were assigned (continued):

	Isotopic mass fractions (-100)	
	Value ³⁾ [g/g]	Uncertainty ⁴⁾ [g/g]
$m(^{233}\text{U})/m(\text{U})$	< 0.0000002	/
$m(^{234}\text{U})/m(\text{U})$	0.0051868	0.0000032
$m(^{235}\text{U})/m(\text{U})$	0.71191	0.00021
$m(^{236}\text{U})/m(\text{U})$	0.000012253	0.000000052
$m(^{238}\text{U})/m(\text{U})$	99.28289	0.00021
	Isotopic amount fractions (-100)	
	Value ³⁾ [mol/mol]	Uncertainty ⁴⁾ [mol/mol]
$n(^{233}\text{U})/n(\text{U})$	< 0.0000002	/
$n(^{234}\text{U})/n(\text{U})$	0.0052752	0.0000032
$n(^{235}\text{U})/n(\text{U})$	0.72096	0.00021
$n(^{236}\text{U})/n(\text{U})$	0.000012356	0.000000052
$n(^{238}\text{U})/n(\text{U})$	99.27376	0.00022
	Molar mass	
	Value ³⁾ [g/mol]	Uncertainty ⁴⁾ [g/mol]
U	238.0288936	0.0000079

³⁾ The derived certified values are calculated from the certified uranium isotope amount ratios and the atomic masses according to G. Audi et al. (The 1993 atomic mass evaluation, Nuclear Physics, A565, 1-65, 1993).

⁴⁾ The 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.

DESCRIPTION OF THE MATERIAL

The IRMM-184 is a uranium isotopic Certified Reference Material (CRM) supplied with isotope amount ratios as certified above. IRMM-184 comes in a flame-sealed glass ampoule containing about 0.004 mol uranium in 5 mL of nitric acid solution. The concentration of nitric acid is 5 mol·L⁻¹.

ANALYTICAL METHODS USED FOR CERTIFICATION

The certified $n(^{235}\text{U})/n(^{238}\text{U})$ ratio was established by mass spectrometric measurements using the MAT511 mass spectrometer. The certified $n(^{234}\text{U})/n(^{238}\text{U})$ and $n(^{236}\text{U})/n(^{238}\text{U})$ ratios were measured using the Triton thermal ionisation mass spectrometer. Compared to the last revision in 2005, the uncertainties for the $n(^{236}\text{U})/n(^{238}\text{U})$ ratios were updated according to the recommendation from ASTM C1832, section 14.6.4, by adding a relative uncertainty contribution of 0.4 % ($k = 2$) for the use of the secondary electron multiplier for measuring ²³⁶U.

SAFETY INFORMATION

The IRMM-184 contains radioactive material. The ampoules should be handled with great care and by experienced personnel in a laboratory suitably equipped for the safe handling of radioactive materials.

INSTRUCTIONS FOR USE AND INTENDED USE

The material is used for calibration of mass spectrometers.

STORAGE

The vials should be stored at + 18 °C ± 5 °C.

However, the European Commission cannot be held responsible for changes that happen during storage of the material at the customer's premises, especially of opened samples.

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Retieseweg 111, B - 2440 Geel (Belgium)

Annex 6. Certificate of IRMM-2025/025



EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Directorate G – Nuclear Safety and Security
G.2 - Standards for Nuclear Safety, Security and Safeguards Unit

CERTIFIED REFERENCE MATERIAL
IRMM-2025
CERTIFICATE OF ANALYSIS

URANYL NITRATE SOLUTION		
	Isotope amount ratio	
	Certified value ¹⁾ [mol/mol]	Uncertainty ²⁾ [mol/mol]
$n(^{234}\text{U})/n(^{238}\text{U})$	0.000122452	0.000000090
$n(^{235}\text{U})/n(^{238}\text{U})$	0.0204356	0.0000055
$n(^{236}\text{U})/n(^{238}\text{U})$	0.000148386	0.000000083
	Isotope amount fraction (x 100)	
	Certified value ³⁾ [mol/mol]	Uncertainty ²⁾ [mol/mol]
$n(^{234}\text{U})/n(\text{U})$	0.0119968	0.0000088
$n(^{235}\text{U})/n(\text{U})$	2.00210	0.00053
$n(^{236}\text{U})/n(\text{U})$	0.0145375	0.0000081
$n(^{238}\text{U})/n(\text{U})$	97.97136	0.00053
	Isotope mass fraction (x 100)	
	Certified value ³⁾⁴⁾ [g/g]	Uncertainty ²⁾ [g/g]
$m(^{234}\text{U})/m(\text{U})$	0.0117977	0.0000086
$m(^{235}\text{U})/m(\text{U})$	1.97732	0.00052
$m(^{236}\text{U})/m(\text{U})$	0.0144188	0.0000081
$m(^{238}\text{U})/m(\text{U})$	97.99646	0.00052
	Molar mass	
	Certified value ³⁾⁴⁾ [g/mol]	Uncertainty ²⁾ [g/mol]
$M(\text{U})$	237.989814	0.000016

All following pages are an integral part of the certificate.
Page 1 of 3

- 1) The certified values are traceable to the International System of units (SI) via IRMM-074/10.
- 2) The 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.
- 3) These values are calculated using the isotope amount ratios and therefore traceable to the SI. The calculation of $n(U)$, $m(U)$ and $M(U)$ includes the contributions from the isotopes ^{234}U , ^{235}U , ^{236}U and ^{238}U .
- 4) These values are calculated using the values listed below from Wang *et al.*, *The AME 2016 atomic mass evaluation*, *Chinese Physics C Vol. 41, No. 3 (2017) 030003*:
- $M(^{234}\text{U}) = 234.0409504 \pm 0.0000024 \text{ g}\cdot\text{mol}^{-1} (k = 2)$
 $M(^{235}\text{U}) = 235.0439282 \pm 0.0000024 \text{ g}\cdot\text{mol}^{-1} (k = 2)$
 $M(^{236}\text{U}) = 236.0455662 \pm 0.0000024 \text{ g}\cdot\text{mol}^{-1} (k = 2)$
 $M(^{238}\text{U}) = 238.0507870 \pm 0.0000032 \text{ g}\cdot\text{mol}^{-1} (k = 2)$

This certificate is valid for 10 years.

Geel, 27 AUG. 2018

Signed:



Dr. Arjan Plompen
European Commission
Joint Research Centre
Directorate G – Nuclear Safety and Security
G.2 - Standards for Nuclear Safety, Security and
Safeguards Unit
Retieseweg 111
B-2440 Geel, Belgium

DESCRIPTION OF THE MATERIAL

IRMM-2025 is a uranyl nitrate solution Isotopic Reference Material as certified above. It is supplied in quartz screw-cap ampoules. The uranium concentration is about 2 mg U / g solution in 5 mL 1M HNO₃.

ANALYTICAL METHODS USED FOR CERTIFICATION

A sample of about 1 gram of the uranium hexafluoride gas reference material IRMM-025 was hydrolysed and converted into uranyl nitrate solution, which was named IRMM-2025. The certified isotopic composition for this solution were established by Thermal Ionisation Mass Spectrometry (TIMS) using the Modified Total Evaporation (MTE) technique according to ASTM C1832. Confirmation measurements were performed on the uranium hexafluoride gas reference material IRMM-025 using Gas Source Mass Spectrometry (GSMS).

SAFETY INFORMATION

The IRMM-2025 contains radioactive material. The ampoule should be handled with great care and by experienced personnel in a laboratory suitably equipped for the safe handling of radioactive materials.

INSTRUCTIONS FOR USE AND INTENDED USE

This reference material is intended to be used as a calibrant for isotope mass spectrometry measurements.

STORAGE

The ampoule should be stored at room temperature in an upright position. However, the European Commission cannot be held responsible for changes that happen during storage of the material at the customer's premises, especially of opened samples.

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NOTE

The certification report for the IRMM-2019 to IRMM-2029 series is available on the internet (<https://crm.jrc.ec.europa.eu>). A paper copy can be obtained from the Joint Research Centre on request.

European Commission – Joint Research Centre
Directorate G – Nuclear Safety and Security
G.2 - Standards for Nuclear Safety, Security and Safeguards Unit
Retieseweg 111, B - 2440 Geel (Belgium)

Annex 7. Certificate of IRMM-058

**CERTIFICATE
SPIKE ISOTOPIC REFERENCE MATERIAL IRMM-058**

$9.013(12) \cdot 10^{-12} \text{ mol } (^{233}\text{U}) \cdot \text{g}^{-1} \text{ (solution)}$

The Spike Isotopic Reference Material is supplied with an isotope amount content of ^{233}U certified as above.

The amount of other uranium isotopes present are related to the ^{233}U content through the following certified amount ratios:

$n(^{234}\text{U})/n(^{233}\text{U}) :$	0.000 352 4(14)
$n(^{235}\text{U})/n(^{233}\text{U}) :$	0.000 004 124(29)
$n(^{236}\text{U})/n(^{233}\text{U}) :$	0.000 000 043 4(14)
$n(^{238}\text{U})/n(^{233}\text{U}) :$	0.000 010 43(21)

This corresponds to an isotopic composition with the following abundances :

amount fraction (-100)		Mass fraction (-100)	
$n(^{233}\text{U})/n(\text{U})$	99.963 32(14)	$m(^{233}\text{U})/m(\text{U})$	99.963 14(14)
$n(^{234}\text{U})/n(\text{U})$	0.035 22(14)	$m(^{234}\text{U})/m(\text{U})$	0.035 37(14)
$n(^{235}\text{U})/n(\text{U})$	0.000 412 3(29)	$m(^{235}\text{U})/m(\text{U})$	0.000 415 8(29)
$n(^{236}\text{U})/n(\text{U})$	0.000 004 34(14)	$m(^{236}\text{U})/m(\text{U})$	0.000 004 40(14)
$n(^{238}\text{U})/n(\text{U})$	0.001 043(21)	$m(^{238}\text{U})/m(\text{U})$	0.001 065(21)

The molar mass of the uranium in this sample is $233.040\ 040\ 4(62) \text{ g} \cdot \text{mol}^{-1}$

From the certified values, the following amount content and mass contents are derived:

$9.016(12) \cdot 10^{-12}$	mol (U) \cdot g ⁻¹ (solution)
$2.100\ 4(29) \cdot 10^{-9}$	g (^{233}U) \cdot g ⁻¹ (solution)
$2.101\ 2(29) \cdot 10^{-9}$	g (U) \cdot g ⁻¹ (solution)

05/01

NOTES

- All uncertainties indicated are expanded uncertainties $U = k \cdot u_c$ where u_c is the combined standard uncertainty estimated following the ISO/BIPM Guide to the Expression of Uncertainty in Measurement. They are given in parentheses and include a coverage factor $k=2$. They apply to the last two digits of the value. The values certified are traceable to the SI.
- The Spike Isotopic Reference Material IRMM-058 comes in a flame-sealed quartz ampoule containing about 0.05 nmol uranium in 5 mL of a chemically stable nitric acid solution.
- The molar masses, used in the calculations, are¹

^{233}U	: 233.039 627 0 (60) g·mol ⁻¹
^{234}U	: 234.040 944 7 (44) g·mol ⁻¹
^{235}U	: 235.043 922 2 (42) g·mol ⁻¹
^{236}U	: 236.045 561 0 (42) g·mol ⁻¹
^{238}U	: 238.050 783 5 (44) g·mol ⁻¹

- The ampoule should be handled with great care and by experienced personnel in a laboratory environment suitably equipped for the safe handling of radioactive materials.
- Using this Spike Isotopic Reference Material, the ^{235}U (or ^{238}U) content in an unknown sample can be determined by Isotope Dilution, through a measurement of the isotope amount ratio $R(B) = n(^{233}\text{U})/n(^{235}\text{U})$ in a blend. It should be computed with the aid of the following equation which enables an easy quantification of the uncertainty sources in the procedure :

$$c(^{235}\text{U}, X) = \frac{R(Y) - R(B)}{R(B) - R(X)} \cdot \frac{1}{R(Y)} \cdot \frac{m(Y)}{m(X)} \cdot c(^{235}\text{U}, Y)$$

$$c(\text{U}, X) = \frac{R(Y) - R(B)}{R(B) - R(X)} \cdot \frac{1 + R(X)}{1 + R(Y)} \cdot \frac{m(Y)}{m(X)} \cdot c(\text{U}, Y)$$

where:

$R(X)$	=	amount ratio $n(^{233}\text{U})/n(^{235}\text{U})$ in the unknown sample material X
$R(Y)$	=	amount ratio $n(^{233}\text{U})/n(^{235}\text{U})$ in the spike material Y
$m(X)$	=	mass of the unknown sample used in the measurement
$m(Y)$	=	mass of the sample of spike solution used in the measurement
$c(^{235}\text{U}, X)$	=	amount content of $^{235}\text{U} \cdot \text{kg}^{-1}$ sample material
$c(^{235}\text{U}, Y)$	=	amount content of $^{235}\text{U} \cdot \text{kg}^{-1}$ spike solution
$c(\text{U}, X)$	=	amount content of U $\cdot \text{kg}^{-1}$ sample material
$c(\text{U}, Y)$	=	amount content of U $\cdot \text{kg}^{-1}$ spike solution.

¹ G. Audi and A.H. Wapstra, The 1993 atomic mass evaluation , Nucl Phys A565 (1993) 1-65.

6. This Spike Isotopic Reference Material is traceable to the SI system in the shortest possible way. Measurements calibrated against these Isotopic reference Materials have therefore the potential of being traceable to the SI

The isotopic measurements were performed by A Alonso-Muñoz and H Kühn by Thermal Ionisation Mass Spectrometry and calibrated by means of synthetic uranium isotope mixtures prepared by W Lycke. Chemical preparation of the samples for isotopic measurements was performed by F Kehoe.

Metrological weighings required in the preparation and certification were performed by F Hendrickx and R Eykens. The ampoulation of this Spike Isotopic Reference Material was accomplished by G Van Baelen, A Held and R Eykens.

The overall co-ordination leading to the establishment, certification and issuance of this Spike Isotopic Reference Material was performed by A Verbruggen.

B-2440 GEEL
May 2001

P Taylor
Head
Isotope Measurements Unit

R Wellum
IRMM Safeguards Coordinator

Annex 8. Certificate of pipette calibration

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<p>Pipette calibration (Eppendorf 1µl 104331) Author: European Commission</p> <p>Calibration of a pipette in compliance to ISO 8655-6:2002 and the NPL Good Practice Guide 69.</p> <p>Ten aliquots of water are transferred into a pre-weighed vial, which is reweighed after each aliquot. The transferred volume is calculated based on the weight increase due to the addition of the aliquots, corrected for evaporation/drifts and the volume displacement, using the so-called Z-factor.</p> <p>Within this GUM Workbench file, the Z-factor is recalculated using the density of water and air estimated from the environmental conditions during the calibration (temperature, ambient pressure and relative humidity). This approach would be more accurate than selecting the relevant factor from the provided table and also accounts for the relative humidity. The density of the air and water are calculated according to the following literature: - Water: Kells equation. Jones f.E., Harris G.L., "ITS-90 Density of water formulation for volumetric standards calibration", J. of Res. of the Nat. Inst. for Stand. And Tech. 97 (1992) pp 335-340 - Air: EURAMET CG18:21011 Annex A1.2</p> <p>Pipette: Eppendorf Nominal volume: 1 µl Serial number: 104331 Tips: Eppendorf epT.I.P.S. 20µl L dualfilter Date of analysis: 24 November 2017 Lab: 206 Balance: XP56, 2011 00172 87 Basis of adjustment: Ex (20 °C)</p> <p>Model Equation:</p> <p>{ Density of Water and Air }</p> $t_w = t_{air}$ $\rho_w = 999.85308 + 6.32693E-2 * t_w - 8.523829E-3 * t_w^2 + 6.943248E-5 * t_w^3 - 3.821216E-7 * t_w^4 + \delta\rho_w;$ $\rho_{air} = (0.348444 * p_{air} - h_{air} * (0.00252 * t_{air} - 0.020582)) / (273.15 + t_{air}) + \delta\rho_{air};$ <p>{ Z correction factor }</p> $Z_{factor} = 1 / (\rho_w - \rho_{air});$ <p>{ Mean pipet volume }</p> $V_{Pipette} = Z_{factor} * (m_{aliquot} - m_{Corr} + \delta_{Bal});$ <p>List of Quantities:</p> <table border="1"> <thead> <tr> <th>Quantity</th> <th>Unit</th> <th>Definition</th> </tr> </thead> <tbody> <tr> <td>$m_{aliquot}$</td> <td></td> <td>Weight of transferred aliquots</td> </tr> <tr> <td>m_{Corr}</td> <td></td> <td>Weight correction for evaporation</td> </tr> <tr> <td>h_{air}</td> <td></td> <td>Relative humidity</td> </tr> <tr> <td>p_{air}</td> <td></td> <td>Ambient air pressure</td> </tr> <tr> <td>t_{air}</td> <td></td> <td>Temperature of air</td> </tr> <tr> <td>t_w</td> <td></td> <td>Temperature of water</td> </tr> <tr> <td>ρ_{air}</td> <td></td> <td>Density of air</td> </tr> <tr> <td>ρ_w</td> <td></td> <td>Density of water</td> </tr> <tr> <td>Z_{factor}</td> <td></td> <td>Correction factor (inverse density)</td> </tr> <tr> <td>δ_{Bal}</td> <td></td> <td>Uncertainty of weighing</td> </tr> </tbody> </table>			Quantity	Unit	Definition	$m_{aliquot}$		Weight of transferred aliquots	m_{Corr}		Weight correction for evaporation	h_{air}		Relative humidity	p_{air}		Ambient air pressure	t_{air}		Temperature of air	t_w		Temperature of water	ρ_{air}		Density of air	ρ_w		Density of water	Z_{factor}		Correction factor (inverse density)	δ_{Bal}		Uncertainty of weighing
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Good practice guide 69	Pipette calibration (Eppendorf 1µl 104331)								
Quantity	Unit	Definition							
$\delta\rho_{air}$		Uncertainty of air density function							
$\delta\rho_w$		Uncertainty of water density function							
$V_{Pipette}$	L	Mean transferred volume							
<p>$m_{aliquot}$: Type A summarized Mean: 0.000839 Standard Deviation of the Mean: 0.000033 Degrees of Freedom: 8</p> <p>Mean weight of the ten transferred aliquots (i.e. $m_x - m_{x-1}$). The data should be entered as "Type A summarized" with a "Standard" uncertainty evaluation, where the standard uncertainty is the standard deviation of the measured weights of aliquots and degrees of freedom is the number of repetitions minus 2 (10-8=8).</p>									
<p>m_{Corr}: Type B rectangular distribution Value: 0 Halfwidth of Limits: 0.000001</p> <p>Correction factor for evaporation or possible drifts. After weighing ten aliquots, the vial is stored for an equal duration after which the weight is recorded. The change of weight is subtracted from the obtained results.</p> <p>Halfwidth=$\delta_{Ba} / 10 * 2$ accounting for two measurements averaged over 10 points.</p>									
<p>h_{air}: Type A Method of observation: Direct Number of observations: 2</p> <table border="1" data-bbox="488 1059 798 1171"> <thead> <tr> <th>No.</th> <th>Observation</th> </tr> </thead> <tbody> <tr> <td>1</td> <td>40.5</td> </tr> <tr> <td>2</td> <td>38.7</td> </tr> </tbody> </table> <p>Arithmetic Mean: 39.600 Standard Deviation: 1.3 Standard Uncertainty: 0.900</p>				No.	Observation	1	40.5	2	38.7
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Date: 03/22/2019 Ver.: 1.0	File: PipetCalibration.SMU		Page 2 of 4						

Good practice guide 69	Pipette calibration (Eppendorf 1µl 104331)																																																																				
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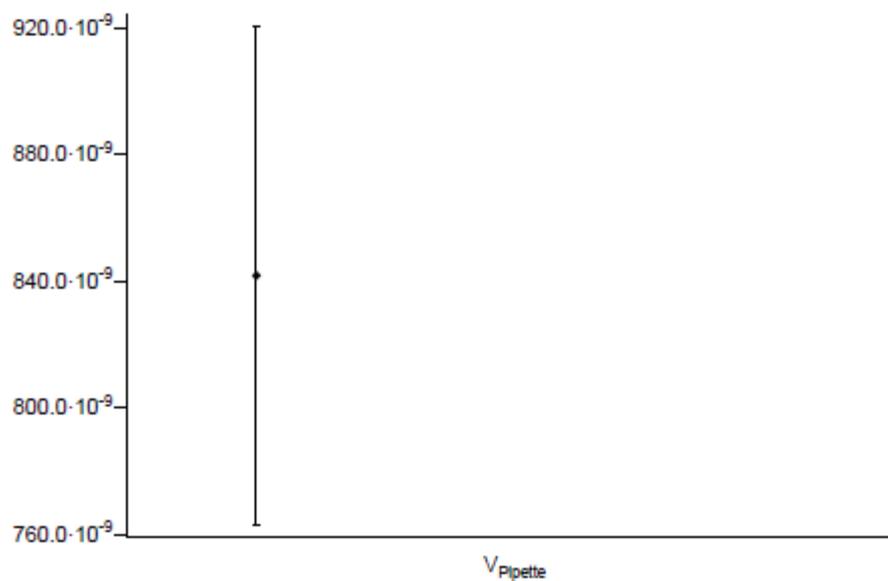
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$V_{Pipette}$: Mean transferred volume

Quantity	Value	Standard Uncertainty	Distribution	Sensitivity Coefficient	Uncertainty Contribution	Index
$m_{aliquot}$	$839.0 \cdot 10^{-6}$	$33.0 \cdot 10^{-6}$	normal	$1.0 \cdot 10^{-3}$	$33 \cdot 10^{-9}$ L	99.2 %
m_{Corr}	0.0	$577 \cdot 10^{-9}$	rectangular	$-1.0 \cdot 10^{-3}$	$-580 \cdot 10^{-12}$ L	0.0 %
h_{air}	39.600	0.900	normal	$-97 \cdot 10^{-15}$	$-88 \cdot 10^{-15}$ L	0.0 %
p_{air}	1007.900	0.300	normal	$1.0 \cdot 10^{-12}$	$300 \cdot 10^{-15}$ L	0.0 %
t_{air}	21.6500	0.0500	normal	$190 \cdot 10^{-12}$	$9.3 \cdot 10^{-12}$ L	0.0 %
δ_{Bal}	0.0	$2.89 \cdot 10^{-6}$	rectangular	$1.0 \cdot 10^{-3}$	$2.9 \cdot 10^{-9}$ L	0.8 %
$\delta_{p_{air}}$	0.0	$200 \cdot 10^{-6}$	normal	$840 \cdot 10^{-12}$	$170 \cdot 10^{-15}$ L	0.0 %
δ_{ρ_w}	0.0	$1.00 \cdot 10^{-3}$	normal	$-840 \cdot 10^{-12}$	$-840 \cdot 10^{-15}$ L	0.0 %
$V_{Pipette}$	$841.8 \cdot 10^{-9}$ L	$33.2 \cdot 10^{-9}$ L				

Results:

Quantity	Value	Expanded Uncertainty	Coverage factor	Coverage
$V_{Pipette}$	$842 \cdot 10^{-9}$ L	$79 \cdot 10^{-9}$ L	2.37	95% (t-table 95.45%)



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Annex 9. Certification of uranium amount per particle in IRMM-2329P

ID-TIMS results for certification of U amount per particle in IRMM-2329P	
<p>ID-TIMS results for certification of U amount per particle in IRMM-2329P Author: European Commission</p> <p>Measurement of the total amount of uranium contained in a single micrometre diameter-sized particle by ID- TIMS.</p> <p>Ten particles (IRMM-2329P) are transferred from the original sample onto a carburized single TIMS filament using micromanipulators under the optical microscope. The particles are dissolved in HNO₃ followed by volumetric addition of a ²³³U spike (IRMM-058). The blend is dried at 0.4 A before measurement, during which the ²³³U/²³⁸U and ²³⁵U/²³⁸U isotope ratios are measured. The ²³³U/²³⁸U isotope ratio is used in combination with the IDMS equation to obtain the uranium mass of single particles.</p> <p>As the addition of the spike causes the major factor to the combined uncertainty of the uranium mass, mostly due to the transferred volume and spike density, the uncertainty due to the spike is added as a separate factor to the combined uncertainty of all acceptable measurements. The relative standard uncertainties due to within-homogeneity (s_{wb}) and between- unit homogeneity (s_{ub}), which were calculated using SoftCRM, were taken into account in final uncertainty budget of the certified values.</p> <p>Element: Uranium Spike: IRMM-058 (²³³U) Sample: IRMM-2329P (²³⁸U) Measurements: T171130, T171214 & T180109 Assumptions:</p> <ul style="list-style-type: none"> - Number of particles: 10 particles (optical microscope) - Mass fractionation factor: 1 - n(²³³U)/n(²³⁸U) in sample: 0 <p>The following samples were rejected:</p> <ul style="list-style-type: none"> - T171130: A1, B3 & C3 - T171214: A1, A2, A3, A5, B2, B3, B4, B5, C2, C4 & C5 - T180109: - <p>Model Equation:</p> <p>{Conversion of isotope ratios }</p> $R_{233/238}^Y = R_{233/233}^Y / R_{238/233}^Y;$ $R_{234/238}^Y = R_{234/233}^Y / R_{238/233}^Y;$ $R_{235/238}^Y = R_{235/233}^Y / R_{238/233}^Y;$ $R_{236/238}^Y = R_{236/233}^Y / R_{238/233}^Y;$ <p>{Sum of the isotope ratios}</p> $\Sigma R_x = R_{233/238}^X + R_{234/238}^X + R_{235/238}^X + R_{236/238}^X + R_{238/238}^X;$ $\Sigma R_y = R_{233/238}^Y + R_{234/238}^Y + R_{235/238}^Y + R_{236/238}^Y + R_{238/238}^Y;$ <p>{Molar mass of sample}</p> $M_x = (R_{233/238}^X * M_{233} + R_{234/238}^X * M_{234} + R_{235/238}^X * M_{235} + R_{236/238}^X * M_{236} + R_{238/238}^X * M_{238}) / \Sigma R_x;$ <p>{ Amount of U added with the spike }</p> $c_y = w_{233} / X_{233} * \rho_y * C_2;$ $n_y = \text{const}(c_y * N_a * V);$	
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ID-TIMS results for certification of U amount per particle in IRMM-2329P		
<p>{ Uranium in blanks }</p> $n_{\text{blank}1} = (R_{233/238Y} - R_{\text{blank}1}) / (R_{\text{blank}1} - R_{233/238X}) * \Sigma R_x / \Sigma R_y * n_y;$ $n_{\text{blank}2} = (R_{233/238Y} - R_{\text{blank}2}) / (R_{\text{blank}2} - R_{233/238X}) * \Sigma R_x / \Sigma R_y * n_y;$ $n_{\text{blank}3} = (R_{233/238Y} - R_{\text{blank}3}) / (R_{\text{blank}3} - R_{233/238X}) * \Sigma R_x / \Sigma R_y * n_y;$ <p>{ Uranium amount per particle }</p> $f_m(R_{\text{blend}}, n_{\text{blank}}) = ((R_{233/238Y} - R_{\text{blend}}) / (R_{\text{blend}} - R_{233/238X}) * \Sigma R_x / \Sigma R_y * n_y - n_{\text{blank}}) / N * C_1;$ $m_{T1T051F2} = f_m(R_{T1T051F2}, n_{\text{blank}1});$ $m_{T1T051F3} = f_m(R_{T1T051F3}, n_{\text{blank}1});$ $m_{T1T051F4} = f_m(R_{T1T051F4}, n_{\text{blank}1});$ $m_{T1T051F5} = f_m(R_{T1T051F5}, n_{\text{blank}1});$ $m_{T1T016F1} = f_m(R_{T1T016F1}, n_{\text{blank}1});$ $m_{T1T016F2} = f_m(R_{T1T016F2}, n_{\text{blank}1});$ $m_{T1T016F4} = f_m(R_{T1T016F4}, n_{\text{blank}1});$ $m_{T1T016F5} = f_m(R_{T1T016F5}, n_{\text{blank}1});$ $m_{T1T078F1} = f_m(R_{T1T078F1}, n_{\text{blank}1});$ $m_{T1T078F2} = f_m(R_{T1T078F2}, n_{\text{blank}1});$ $m_{T1T078F4} = f_m(R_{T1T078F4}, n_{\text{blank}1});$ $m_{T1T078F5} = f_m(R_{T1T078F5}, n_{\text{blank}1});$ $m_{T2T069F4} = f_m(R_{T2T069F4}, n_{\text{blank}2});$ $m_{T2T020F1} = f_m(R_{T2T020F1}, n_{\text{blank}2});$ $m_{T2T102F1} = f_m(R_{T2T102F1}, n_{\text{blank}2});$ $m_{T2T102F3} = f_m(R_{T2T102F3}, n_{\text{blank}2});$ $m_{T3T069F1} = f_m(R_{T3T069F1}, n_{\text{blank}3});$ $m_{T3T069F2} = f_m(R_{T3T069F2}, n_{\text{blank}3});$ $m_{T3T069F3} = f_m(R_{T3T069F3}, n_{\text{blank}3});$ $m_{T3T069F4} = f_m(R_{T3T069F4}, n_{\text{blank}3});$ $m_{T3T069F5} = f_m(R_{T3T069F5}, n_{\text{blank}3});$ $m_{T3T020F1} = f_m(R_{T3T020F1}, n_{\text{blank}3});$ $m_{T3T020F2} = f_m(R_{T3T020F2}, n_{\text{blank}3});$ $m_{T3T020F3} = f_m(R_{T3T020F3}, n_{\text{blank}3});$ $m_{T3T020F4} = f_m(R_{T3T020F4}, n_{\text{blank}3});$ $m_{T3T020F5} = f_m(R_{T3T020F5}, n_{\text{blank}3});$ $m_{T3T102F1} = f_m(R_{T3T102F1}, n_{\text{blank}3});$ $m_{T3T102F2} = f_m(R_{T3T102F2}, n_{\text{blank}3});$ $m_{T3T102F3} = f_m(R_{T3T102F3}, n_{\text{blank}3});$ $m_{T3T102F4} = f_m(R_{T3T102F4}, n_{\text{blank}3});$ $m_{T3T102F5} = f_m(R_{T3T102F5}, n_{\text{blank}3});$ $n_{\text{char}} = \text{average}(m_{T1T051F2}, m_{T1T051F3}, m_{T1T051F4}, m_{T1T051F5}, m_{T1T016F1}, m_{T1T016F2}, m_{T1T016F4}, m_{T1T016F5}, m_{T1T078F1}, m_{T1T078F2}, m_{T1T078F4}, m_{T1T078F5}, m_{T2T069F4}, m_{T2T020F1}, m_{T2T102F1}, m_{T2T102F3}, m_{T3T069F1},$		
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$m_{T11078F1}, m_{T11078F2}, m_{T11078F4}, m_{T11078F5}, m_{T2089F4}, m_{T2020F1}, m_{T2102F1}, m_{T2102F3}, m_{T3089F1},$
 $m_{T3089F2}, m_{T3089F3}, m_{T3089F4}, m_{T3089F5}, m_{T3020F1}, m_{T3020F2}, m_{T3020F3}, m_{T3020F4}, m_{T3020F5},$
 $m_{T3102F1}, m_{T3102F2}, m_{T3102F3}, m_{T3102F4}, m_{T3102F5}$;

{ Combination of uncertainty }

$$\delta n_{ip} = c_y \cdot N_A \cdot V / \text{const}(n_y);$$

$$\delta x_{bb}; \delta x_{swb};$$

$$n_{char,combined} = n_{char} \cdot \delta n_{ip};$$

$$m_{char,combined} = n_{char,combined} \cdot M_x \cdot C_3;$$

$$n_{CRM} = n_{char} \cdot \delta n_{ip} \cdot \delta x_{bb} \cdot \delta x_{swb};$$

$$m_{CRM} = n_{CRM} \cdot M_x \cdot C_3;$$

$$N_{CRM} = n_{CRM} / C_1 \cdot N_A;$$

List of Quantities:

Quantity	Unit	Definition
C_1	fmol/mol	Conversion from mol to fmol
C_2	mL/L	Conversion from l to ml
C_3	pg/fg	Conversion from fg to pg
c_y	mol/L	Uranium concentration of spike
M_{233}	g/mol	Molar mass of U-233
M_{234}	g/mol	Molar mass of U-234
M_{235}	g/mol	Molar mass of U-235
M_{236}	g/mol	Molar mass of U-236
M_{238}	g/mol	Molar mass of U-238
$m_{T11018F1}$	fmol	Uranium mass per particle for turret 1, sample 16, filament 1
$m_{T11018F2}$	fmol	Uranium mass per particle for turret 1, sample 16, filament 2
$m_{T11018F4}$	fmol	Uranium mass per particle for turret 1, sample 16, filament 4
$m_{T11018F5}$	fmol	Uranium mass per particle for turret 1, sample 16, filament 5
$m_{T11051F2}$	fmol	Uranium mass per particle for turret 1, sample 51, filament 2
$m_{T11051F3}$	fmol	Uranium mass per particle for turret 1, sample 51, filament 3
$m_{T11051F4}$	fmol	Uranium mass per particle for turret 1, sample 51, filament 4
$m_{T11051F5}$	fmol	Uranium mass per particle for turret 1, sample 51, filament 5
$m_{T11078F1}$	fmol	Uranium mass per particle for turret 1, sample 78, filament 1
$m_{T11078F2}$	fmol	Uranium mass per particle for turret 1, sample 78, filament 2
$m_{T11078F4}$	fmol	Uranium mass per particle for turret 1, sample 78, filament 4
$m_{T11078F5}$	fmol	Uranium mass per particle for turret 1, sample 78, filament 5
$m_{T2020F1}$	fmol	Uranium mass per particle for turret 2, sample 20, filament 1
$m_{T2089F4}$	fmol	Uranium mass per particle for turret 2, sample 69, filament 4
$m_{T2102F1}$	fmol	Uranium mass per particle for turret 2, sample 102, filament 1
$m_{T2102F3}$	fmol	Uranium mass per particle for turret 2, sample 102, filament 3
$m_{T3020F1}$	fmol	Uranium mass per particle for turret 3, sample 20, filament 1

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Quantity	Unit	Definition
$m_{T3T020F2}$	fmol	Uranium mass per particle for turret 3, sample 20, filament 2
$m_{T3T020F3}$	fmol	Uranium mass per particle for turret 3, sample 20, filament 3
$m_{T3T020F4}$	fmol	Uranium mass per particle for turret 3, sample 20, filament 4
$m_{T3T020F5}$	fmol	Uranium mass per particle for turret 3, sample 20, filament 5
$m_{T3T069F1}$	fmol	Uranium mass per particle for turret 3, sample 69, filament 1
$m_{T3T069F2}$	fmol	Uranium mass per particle for turret 3, sample 69, filament 2
$m_{T3T069F3}$	fmol	Uranium mass per particle for turret 3, sample 69, filament 3
$m_{T3T069F4}$	fmol	Uranium mass per particle for turret 3, sample 69, filament 4
$m_{T3T069F5}$	fmol	Uranium mass per particle for turret 3, sample 69, filament 5
$m_{T3T102F1}$	fmol	Uranium mass per particle for turret 3, sample 102, filament 1
$m_{T3T102F2}$	fmol	Uranium mass per particle for turret 3, sample 102, filament 2
$m_{T3T102F3}$	fmol	Uranium mass per particle for turret 3, sample 102, filament 3
$m_{T3T102F4}$	fmol	Uranium mass per particle for turret 3, sample 102, filament 4
$m_{T3T102F5}$	fmol	Uranium mass per particle for turret 3, sample 102, filament 5
n_{char}	fmol	Average uranium mass per particle
M_x	g/mol	Molar mass of sample
N		Number of particles
N_a		Number of aliquots of spike added
n_{blank1}	mol	Amount of uranium in blank 1
n_{blank2}	mol	Amount of uranium in blank 2
n_{blank3}	mol	Amount of uranium in blank 3
n_y	mol	Amount of uranium added with spike
$R_{233/233Y}$		U-233/U-233 ratio of spike
$R_{234/233Y}$		U-234/U-233 ratio of spike
$R_{235/233Y}$		U-235/U-233 ratio of spike
$R_{236/233Y}$		U-236/U-233 ratio of spike
$R_{238/233Y}$		U-238/U-233 ratio of spike
$R_{233/238Y}$		U-233/U-238 ratio of spike
$R_{234/238Y}$		U-234/U-238 ratio of spike
$R_{235/238Y}$		U-235/U-238 ratio of spike
$R_{236/238Y}$		U-236/U-238 ratio of spike
$R_{238/238Y}$		U-238/U-238 ratio of spike
$R_{233/238X}$		U-233/U-238 ratio of sample
$R_{234/238X}$		U-234/U-238 ratio of sample
$R_{235/238X}$		U-235/U-238 ratio of sample
$R_{236/238X}$		U-236/U-238 ratio of sample
$R_{238/238X}$		U-238/U-238 ratio of sample
R_{blank1}		Measured 233/238 ratio for blank 1

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Quantity	Unit	Definition
R _{blank3}		Measured 233/238 ratio for blank 3
R _{T1T016F1}		Measured 233/238 ratio for turret 1, sample 16, filament 1
R _{T1T016F2}		Measured 233/238 ratio for turret 1, sample 16, filament 2
R _{T1T016F4}		Measured 233/238 ratio for turret 1, sample 16, filament 4
R _{T1T016F5}		Measured 233/238 ratio for turret 1, sample 16, filament 5
R _{T1T051F2}		Measured 233/238 ratio for turret 1, sample 51, filament 2
R _{T1T051F3}		Measured 233/238 ratio for turret 1, sample 51, filament 3
R _{T1T051F4}		Measured 233/238 ratio for turret 1, sample 51, filament 4
R _{T1T051F5}		Measured 233/238 ratio for turret 1, sample 51, filament 5
R _{T1T078F1}		Measured 233/238 ratio for turret 1, sample 78, filament 1
R _{T1T078F2}		Measured 233/238 ratio for turret 1, sample 78, filament 2
R _{T1T078F4}		Measured 233/238 ratio for turret 1, sample 78, filament 4
R _{T1T078F5}		Measured 233/238 ratio for turret 1, sample 78, filament 5
R _{T2T020F1}		Measured 233/238 ratio for turret 2, sample 20, filament 1
R _{T2T069F4}		Measured 233/238 ratio for turret 2, sample 69, filament 4
R _{T2T102F1}		Measured 233/238 ratio for turret 2, sample 102, filament 1
R _{T2T102F3}		Measured 233/238 ratio for turret 2, sample 102, filament 3
R _{T3T020F1}		Measured 233/238 ratio for turret 3, sample 20, filament 1
R _{T3T020F2}		Measured 233/238 ratio for turret 3, sample 20, filament 2
R _{T3T020F3}		Measured 233/238 ratio for turret 3, sample 20, filament 3
R _{T3T020F4}		Measured 233/238 ratio for turret 3, sample 20, filament 4
R _{T3T020F5}		Measured 233/238 ratio for turret 3, sample 20, filament 5
R _{T3T069F1}		Measured 233/238 ratio for turret 3, sample 69, filament 1
R _{T3T069F2}		Measured 233/238 ratio for turret 3, sample 69, filament 2
R _{T3T069F3}		Measured 233/238 ratio for turret 3, sample 69, filament 3
R _{T3T069F4}		Measured 233/238 ratio for turret 3, sample 69, filament 4
R _{T3T069F5}		Measured 233/238 ratio for turret 3, sample 69, filament 5
R _{T3T102F1}		Measured 233/238 ratio for turret 3, sample 102, filament 1
R _{T3T102F2}		Measured 233/238 ratio for turret 3, sample 102, filament 2
R _{T3T102F3}		Measured 233/238 ratio for turret 3, sample 102, filament 3
R _{T3T102F4}		Measured 233/238 ratio for turret 3, sample 102, filament 4
R _{T3T102F5}		Measured 233/238 ratio for turret 3, sample 102, filament 5
V	L	Volume of spike aliquots
w _{233Y}	mol/g	U-233 content of spike
X _{233Y}		U-233 amount fraction in spike
ρ _y	g/mL	Density of spike
ΣR _x		Sum of isotope ratios of sample
ΣR _y		Sum of isotope ratios of spike
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Quantity	Unit	Definition
ΣR_y		Sum of isotope ratios of spike
N_A	mol ⁻¹	Avogadro's number
δn_p		Uncertainty contribution due to spike addition
δX_{bb}		Uncertainty contribution due to between-bottle inhomogeneity
δX_{swc}		Uncertainty contribution due to short-term instability
$n_{char,combined}$	fmol	Average uranium amount per particle with combined uncertainty for characterisation study
$m_{char,combined}$	pg	Average uranium mass per particle with combined uncertainty for characterisation study
n_{CRM}	fmol	Uranium amount per particle of certified particles
m_{CRM}	pg	Uranium mass per particle of certified particles
N_{CRM}		Number of atoms per particle
C₁ :	Constant Value: 1·10 ¹⁵ fmol/mol	
	Conversion from mol to fmol, constant	
C₂ :	Constant Value: 1·10 ³ mL/L	
	Conversion from l to ml, constant	
C₃ :	Constant Value: 1·10 ⁻³ pg/fg	
	Conversion from fg to pg, constant	
M₂₃₃ :	Type B normal distribution Value: 233.0396344 g/mol Expanded Uncertainty: 0.0000024 g/mol Coverage Factor: 1	
	Value and uncertainty from AME2016 Chinese Physics C 41 (2017) 030003 https://www-nds.iaea.org/amdc/	
M₂₃₄ :	Type B normal distribution Value: 234.0409504 g/mol Expanded Uncertainty: 0.0000012 g/mol Coverage Factor: 1	
	Value and uncertainty from AME2016 Chinese Physics C 41 (2017) 030003 https://www-nds.iaea.org/amdc/	
M₂₃₅ :	Type B normal distribution Value: 235.0439282 g/mol Expanded Uncertainty: 0.0000012 g/mol Coverage Factor: 1	
	Value and uncertainty from AME2016 Chinese Physics C 41 (2017) 030003 https://www-nds.iaea.org/amdc/	
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ID-TIMS results for certification of U amount per particle in IRMM-2329P		
M₂₃₆ :	Type B normal distribution Value: 236.0455662 g/mol Expanded Uncertainty: 0.0000012 g/mol Coverage Factor: 1	
Value and uncertainty from AME2016 Chinese Physics C 41 (2017) 030003 https://www-nds.iaea.org/amdc/		
M₂₃₈ :	Type B normal distribution Value: 238.0507870 g/mol Expanded Uncertainty: 0.0000016 g/mol Coverage Factor: 1	
Value and uncertainty from AME2016 Chinese Physics C 41 (2017) 030003 https://www-nds.iaea.org/amdc/		
N :	Constant Value: 10	
Number of analysed particles, taken as constant		
N_s :	Constant Value: 3	
Number of transferred aliquots of spike, taken as constant		
R_{233/233Y} :	Constant Value: 1	
233/233 ratio is constant (=1)		
R_{234/233Y} :	Type B normal distribution Value: 0.0003524 Expanded Uncertainty: 0.0000014 Coverage Factor: 2	
Isotope amount ratio from certificate of spike (IRMM-058)		
R_{235/233Y} :	Type B normal distribution Value: 0.000004124 Expanded Uncertainty: 0.000000029 Coverage Factor: 2	
Isotope amount ratio from certificate of spike (IRMM-058)		
R_{236/233Y} :	Type B normal distribution Value: 0.0000000434 Expanded Uncertainty: 0.0000000014 Coverage Factor: 2	
Isotope amount ratio from certificate of spike (IRMM-058)		
R_{238/233Y} :	Type B normal distribution Value: 0.00001043 Expanded Uncertainty: 0.000000021 Coverage Factor: 2	
Isotope amount ratio from certificate of spike (IRMM-058)		
R_{238/238Y} :	Constant Value: 1	
238/238 ratio is constant (=1)		
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R_{233/238}x:	Constant Value: 0	
U-233/U-238 isotope ratio of sample taken as constant =0, as not given on certificate		
R_{234/238}x:	Import Filename: IRMM2329P_isotopics_CVe.smu Symbol: R _{234/238}	
Isotope ratio from certificate of IRMM-2329 (base solution) #3867		
R_{235/238}x:	Import Filename: IRMM2329P_isotopics_CVe.smu Symbol: R _{235/238}	
The ²³⁵ U/ ²³⁸ U isotope amount ratio of the sample has been taken from the measurement certificate #3867		
R_{236/238}x:	Import Filename: IRMM2329P_isotopics_CVe.smu Symbol: R _{236/238}	
Isotope ratio from certificate of IRMM-2329 (base solution) #3867		
R_{238/238}x:	Constant Value: 1	
238/238 ratio is constant (=1)		
R_{blank1}:	Type B normal distribution Value: 77 Expanded Uncertainty: 2 Coverage Factor: 2	
Measured isotope ratio taken from internal test report #4055		
R_{blank2}:	Type B normal distribution Value: 57 Expanded Uncertainty: 4 Coverage Factor: 2	
Measured isotope ratio taken from internal test report #4055		
R_{blank3}:	Type B normal distribution Value: 28 Expanded Uncertainty: 2 Coverage Factor: 1	
Measured isotope ratio taken from internal test report #4055		
R_{T1T016F1}:	Type B normal distribution Value: 0.15449 Expanded Uncertainty: 0.00049 Coverage Factor: 2	
Measured isotope ratio taken from internal test report #4055		
R_{T1T016F2}:	Type B normal distribution Value: 0.13837 Expanded Uncertainty: 0.00033 Coverage Factor: 2	
Measured isotope ratio taken from internal test report #4055		
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R_{T1T018F4}	Type B normal distribution Value: 0.13402 Expanded Uncertainty: 0.00036 Coverage Factor: 2	
Measured isotope ratio taken from internal test report #4055		
R_{T1T018F5}	Type B normal distribution Value: 0.17375 Expanded Uncertainty: 0.00074 Coverage Factor: 2	
Measured isotope ratio taken from internal test report #4055		
R_{T1T051F2}	Type B normal distribution Value: 0.16349 Expanded Uncertainty: 0.00041 Coverage Factor: 2	
Measured isotope ratio taken from internal test report #4055		
R_{T1T051F3}	Type B normal distribution Value: 0.15950 Expanded Uncertainty: 0.00061 Coverage Factor: 2	
Measured isotope ratio taken from internal test report #4055		
R_{T1T051F4}	Type B normal distribution Value: 0.15669 Expanded Uncertainty: 0.00069 Coverage Factor: 2	
Measured isotope ratio taken from internal test report #4055		
R_{T1T051F5}	Type B normal distribution Value: 0.17027 Expanded Uncertainty: 0.00089 Coverage Factor: 2	
Measured isotope ratio taken from internal test report #4055		
R_{T1T078F1}	Type B normal distribution Value: 0.14574 Expanded Uncertainty: 0.00023 Coverage Factor: 2	
Measured isotope ratio taken from internal test report #4055		
R_{T1T078F2}	Type B normal distribution Value: 0.14352 Expanded Uncertainty: 0.00048 Coverage Factor: 2	
Measured isotope ratio taken from internal test report #4055		
R_{T1T078F4}	Type B normal distribution Value: 0.14932 Expanded Uncertainty: 0.00035 Coverage Factor: 2	
Measured isotope ratio taken from internal test report #4055		
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R_{T1T078F5}	Type B normal distribution Value: 0.14474 Expanded Uncertainty: 0.00046 Coverage Factor: 2	
Measured isotope ratio taken from internal test report #4055		
R_{T2T020F1}	Type B normal distribution Value: 0.1760 Expanded Uncertainty: 0.0012 Coverage Factor: 2	
Measured isotope ratio taken from internal test report #4055		
R_{T2T089F4}	Type B normal distribution Value: 0.1462 Expanded Uncertainty: 0.0022 Coverage Factor: 2	
Measured isotope ratio taken from internal test report #4055		
R_{T2T102F1}	Type B normal distribution Value: 0.1612 Expanded Uncertainty: 0.0022 Coverage Factor: 2	
Measured isotope ratio taken from internal test report #4055		
R_{T2T102F3}	Type B normal distribution Value: 0.17040 Expanded Uncertainty: 0.00088 Coverage Factor: 2	
Measured isotope ratio taken from internal test report #4055		
R_{T3T020F1}	Type B normal distribution Value: 0.17624 Expanded Uncertainty: 0.00086 Coverage Factor: 2	
Measured isotope ratio taken from internal test report #4055		
R_{T3T020F2}	Type B normal distribution Value: 0.17178 Expanded Uncertainty: 0.00075 Coverage Factor: 2	
Measured isotope ratio taken from internal test report #4055		
R_{T3T020F3}	Type B normal distribution Value: 0.19924 Expanded Uncertainty: 0.00034 Coverage Factor: 2	
Measured isotope ratio taken from internal test report #4055		
R_{T3T020F4}	Type B normal distribution Value: 0.19101 Expanded Uncertainty: 0.00081 Coverage Factor: 2	
Measured isotope ratio taken from internal test report #4055		
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R_{T3T020F5}	Type B normal distribution Value: 0.1843 Expanded Uncertainty: 0.0011 Coverage Factor: 2	
Measured isotope ratio taken from internal test report #4055		
R_{T3T069F1}	Type B normal distribution Value: 0.16801 Expanded Uncertainty: 0.00080 Coverage Factor: 2	
Measured isotope ratio taken from internal test report #4055		
R_{T3T069F2}	Type B normal distribution Value: 0.16050 Expanded Uncertainty: 0.00054 Coverage Factor: 2	
Measured isotope ratio taken from internal test report #4055		
R_{T3T069F3}	Type B normal distribution Value: 0.15317 Expanded Uncertainty: 0.00058 Coverage Factor: 2	
Measured isotope ratio taken from internal test report #4055		
R_{T3T069F4}	Type B normal distribution Value: 0.15947 Expanded Uncertainty: 0.00074 Coverage Factor: 2	
Measured isotope ratio taken from internal test report #4055		
R_{T3T069F5}	Type B normal distribution Value: 0.16186 Expanded Uncertainty: 0.00059 Coverage Factor: 2	
Measured isotope ratio taken from internal test report #4055		
R_{T3T102F1}	Type B normal distribution Value: 0.15596 Expanded Uncertainty: 0.00080 Coverage Factor: 2	
Measured isotope ratio taken from internal test report #4055		
R_{T3T102F2}	Type B normal distribution Value: 0.16231 Expanded Uncertainty: 0.00050 Coverage Factor: 2	
Measured isotope ratio taken from internal test report #4055		
R_{T3T102F3}	Type B normal distribution Value: 0.16483 Expanded Uncertainty: 0.00060 Coverage Factor: 2	
Measured isotope ratio taken from internal test report #4055		
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ID-TIMS results for certification of U amount per particle in IRMM-2329P		
R_{T3T102F4} :	Type B normal distribution Value: 0.16401 Expanded Uncertainty: 0.00052 Coverage Factor: 2	
Measured isotope ratio taken from internal test report #4055		
R_{T3T102F5} :	Type B normal distribution Value: 0.16828 Expanded Uncertainty: 0.00053 Coverage Factor: 2	
Measured isotope ratio taken from internal test report #4055		
V :	Import Filename: PipetCalibration_CVe.SMU Symbol: V _{Pipette}	
Uncertainty of aliquot volume taken from pipette calibration of the used pipet. Calculated in external GUM Workbench file		
w_{233Y} :	Type B normal distribution Value: 9.013·10 ⁻¹² mol/g Expanded Uncertainty: 0.012·10 ⁻¹² mol/g Coverage Factor: 2	
U-233 amount content taken from certificate of spike (IRMM-058)		
X_{233Y} :	Type B normal distribution Value: 0.9996332 Expanded Uncertainty: 0.0000014 Coverage Factor: 2	
U-233 amount fraction taken from certificate of spike (IRMM-058)		
ρ_y :	Import Filename: DensityHNO3.smu Symbol: ρ	
Density of spike (IRMM-058) calculated according to Cauchetier1985 (FR) & Sakurai1996 (EN), assuming 2.1 ng/g U in 1 mol/L HNO ₃ . Calculated in external GUM Workbench file		
N_A :	Type B normal distribution Value: 6.022140857·10 ²³ mol ⁻¹ Expanded Uncertainty: 0.000000074·10 ²³ mol ⁻¹ Coverage Factor: 1	
Avogadro's constant taken from NIST https://physics.nist.gov/cgi-bin/cuu/Value?na		
δx_{bb} :	Import from Excel Filename: Homogeneity U amount per particle_IRMM2329P_SoftCRM.xls Worksheet: ANOVA Value Cell: I6 = 1.0000 Standarduncertainty Cell: D6 = 0.0550	
Between-bottle inhomogeneity calculated using ANOVA single factor analysis in MS Excel. File: Homogeneity U amount per particle _{IRMM2329P} SoftCRM.xls		
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ID-TIMS results for certification of U amount per particle in IRMM-2329P

δx_{sub} : Import from Excel
 Filename: Homogeneity U amount per particle_IRMM2329P_SoftCRM.xls
 Worksheet: ANOVA
 Value Cell: I6 = 1.0000
 Standarduncertainty Cell: F6 = 0.0550

File: Homogeneity U amount per particle_IRMM2329P_SoftCRM.xls

Interim Results:

Quantity	Value	Standard Uncertainty
c_y	$9.3152 \cdot 10^{-9}$ mol/L	$19.4 \cdot 10^{-12}$ mol/L
M_x	237.9508465 g/mol	$16.9 \cdot 10^{-6}$ g/mol
$R_{233/238}^y$	95877	965
$R_{234/238}^y$	33.787	0.347
$R_{235/238}^y$	0.39540	$4.22 \cdot 10^{-3}$
$R_{236/238}^y$	$4.1611 \cdot 10^{-3}$	$79.1 \cdot 10^{-6}$
ΣR_x	1.03427303	$6.00 \cdot 10^{-6}$
ΣR_y	95912	966

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ID-TIMS results for certification of U amount per particle in
IRMM-2329P

Uncertainty Budgets:

n_{char} : Average uranium mass per particle

Quantity	Value	Standard Uncertainty	Distribution	Sensitivity Coefficient	Uncertainty Contribution	Index
C ₁	1.0·10 ¹⁵ fmol/mol					
C ₂	1000.0 mL/L					
N	10.0					
N _a	3.0					
R _{233/233Y}	1.0					
R _{234/233Y}	352.400·10 ⁻⁶	700·10 ⁻⁹	normal	-15	-11·10 ⁻⁶ fmol	0.0 %
R _{235/233Y}	4.1240·10 ⁻⁶	14.5·10 ⁻⁹	normal	-15	-220·10 ⁻⁹ fmol	0.0 %
R _{236/233Y}	43.400·10 ⁻⁶	700·10 ⁻¹²	normal	-15	-11·10 ⁻⁹ fmol	0.0 %
R _{238/233Y}	10.430·10 ⁻⁶	105·10 ⁻⁹	normal	-15	-1.6·10 ⁻⁶ fmol	0.0 %
R _{238/238Y}	1.0					
R _{233/238X}	0.0					
R _{234/238X}	340.8250·10 ⁻⁶	93.0·10 ⁻⁹	normal	15	1.4·10 ⁻⁵ fmol	0.0 %
R _{235/238X}	0.03390200	6.00·10 ⁻⁶	normal	15	87·10 ⁻⁵ fmol	0.0 %
R _{236/238X}	30.2070·10 ⁻⁶	62.0·10 ⁻⁹	normal	15	900·10 ⁻⁹ fmol	0.0 %
R _{238/238X}	1.0					
R _{blank1}	77.00	1.00	normal	160·10 ⁻⁶	160·10 ⁻⁶ fmol	0.0 %
R _{blank2}	57.00	2.00	normal	97·10 ⁻⁶	190·10 ⁻⁶ fmol	0.0 %
R _{blank3}	28.00	2.00	normal	not valid!	3.0·10 ⁻³ fmol	14.5 %
R _{T1T016F1}	0.154490	245·10 ⁻⁶	normal	-3.3	-810·10 ⁻⁶ fmol	1.0 %
R _{T1T016F2}	0.138370	165·10 ⁻⁶	normal	-4.1	-680·10 ⁻⁶ fmol	0.7 %
R _{T1T016F4}	0.134020	180·10 ⁻⁶	normal	-4.4	-790·10 ⁻⁶ fmol	1.0 %
R _{T1T016F5}	0.173750	370·10 ⁻⁶	normal	-2.6	-960·10 ⁻⁶ fmol	1.5 %
R _{T1T051F2}	0.163490	205·10 ⁻⁶	normal	-2.9	-600·10 ⁻⁶ fmol	0.6 %
R _{T1T051F3}	0.159500	305·10 ⁻⁶	normal	-3.1	-940·10 ⁻⁶ fmol	1.4 %
R _{T1T051F4}	0.156690	345·10 ⁻⁶	normal	-3.2	-1.1·10 ⁻³ fmol	1.9 %
R _{T1T051F5}	0.170270	445·10 ⁻⁶	normal	-2.7	-1.2·10 ⁻³ fmol	2.3 %
R _{T1T078F1}	0.145740	115·10 ⁻⁶	normal	-3.7	-420·10 ⁻⁶ fmol	0.3 %
R _{T1T078F2}	0.143520	240·10 ⁻⁶	normal	-3.8	-910·10 ⁻⁶ fmol	1.3 %
R _{T1T078F4}	0.149320	175·10 ⁻⁶	normal	-3.5	-620·10 ⁻⁶ fmol	0.6 %
R _{T1T078F5}	0.144740	230·10 ⁻⁶	normal	-3.7	-860·10 ⁻⁶ fmol	1.2 %
R _{T2T020F1}	0.176000	600·10 ⁻⁶	normal	-2.5	-1.5·10 ⁻³ fmol	3.7 %
R _{T2T069F4}	0.14620	1.10·10 ⁻³	normal	-3.7	-4.0·10 ⁻³ fmol	26.0 %
R _{T2T102F1}	0.16120	1.10·10 ⁻³	normal	-3.0	-3.3·10 ⁻³ fmol	17.6 %
R _{T2T102F3}	0.170400	440·10 ⁻⁶	normal	-2.7	-1.2·10 ⁻³ fmol	2.3 %
R _{T3T020F1}	0.176240	430·10 ⁻⁶	normal	-2.5	-1.1·10 ⁻³ fmol	1.9 %

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ID-TIMS results for certification of U amount per particle in IRMM-2329P

Quantity	Value	Standard Uncertainty	Distribution	Sensitivity Coefficient	Uncertainty Contribution	Index
R _{T3T020F2}	0.171780	375·10 ⁻⁶	normal	-2.7	-1.0·10 ⁻³ fmol	1.6 %
R _{T3T020F3}	0.199240	170·10 ⁻⁶	normal	-2.0	-340·10 ⁻⁶ fmol	0.2 %
R _{T3T020F4}	0.191010	405·10 ⁻⁶	normal	-2.2	-870·10 ⁻⁶ fmol	1.2 %
R _{T3T020F5}	0.184300	550·10 ⁻⁶	normal	-2.3	-1.3·10 ⁻³ fmol	2.6 %
R _{T3T069F1}	0.168010	400·10 ⁻⁶	normal	-2.8	-1.1·10 ⁻³ fmol	2.0 %
R _{T3T069F2}	0.160500	270·10 ⁻⁶	normal	-3.0	-820·10 ⁻⁶ fmol	1.1 %
R _{T3T069F3}	0.153170	290·10 ⁻⁶	normal	-3.3	-970·10 ⁻⁶ fmol	1.5 %
R _{T3T069F4}	0.159470	370·10 ⁻⁶	normal	-3.1	-1.1·10 ⁻³ fmol	2.1 %
R _{T3T069F5}	0.161860	295·10 ⁻⁶	normal	-3.0	-880·10 ⁻⁶ fmol	1.2 %
R _{T3T102F1}	0.155960	400·10 ⁻⁶	normal	-3.2	-1.3·10 ⁻³ fmol	2.7 %
R _{T3T102F2}	0.162310	250·10 ⁻⁶	normal	-3.0	-740·10 ⁻⁶ fmol	0.9 %
R _{T3T102F3}	0.164830	300·10 ⁻⁶	normal	-2.9	-870·10 ⁻⁶ fmol	1.2 %
R _{T3T102F4}	0.164010	260·10 ⁻⁶	normal	-2.9	-760·10 ⁻⁶ fmol	0.9 %
R _{T3T102F5}	0.168280	265·10 ⁻⁶	normal	-2.8	-730·10 ⁻⁶ fmol	0.9 %
V	841.8·10 ⁻⁹ L	33.2·10 ⁻⁹ L		0.0	0.0 fmol	0.0 %
w _{233Y}	9.01300·10 ⁻¹² mol/g	6.00·10 ⁻¹⁵ mol/g	normal	0.0	0.0 fmol	0.0 %
X _{233Y}	0.999633200	700·10 ⁻⁹	normal	0.0	0.0 fmol	0.0 %
ρ _y	1.03315 g/mL	2.04·10 ⁻³ g/mL		0.0	0.0 fmol	0.0 %
n _{char}	15.05166 fmol	7.91·10 ⁻³ fmol				

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ID-TIMS results for certification of U amount per particle in IRMM-2329P						
n_{blank} 1: Amount of uranium in blank 1						
Quantity	Value	Standard Uncertainty	Distribution	Sensitivity Coefficient	Uncertainty Contribution	Index
C ₂	1000.0 mL/L					
N _a	3.0					
R _{233/233Y}	1.0					
R _{234/233Y}	352.400·10 ⁻⁶	700·10 ⁻⁹	normal	-320·10 ⁻¹⁸	-220·10 ⁻²⁴ mol	0.0 %
R _{235/233Y}	4.1240·10 ⁻⁶	14.5·10 ⁻⁹	normal	-320·10 ⁻¹⁸	-4.6·10 ⁻²⁴ mol	0.0 %
R _{238/233Y}	43.400·10 ⁻⁹	700·10 ⁻¹²	normal	-320·10 ⁻¹⁸	-220·10 ⁻²⁷ mol	0.0 %
R _{238/233Y}	10.430·10 ⁻⁶	105·10 ⁻⁹	normal	-25·10 ⁻¹⁵	-2.6·10 ⁻²¹ mol	0.0 %
R _{238/238Y}	1.0					
R _{233/238X}	0.0					
R _{234/238X}	340.8250·10 ⁻⁶	93.0·10 ⁻⁹	normal	310·10 ⁻¹⁸	28·10 ⁻²⁴ mol	0.0 %
R _{235/238X}	0.03390200	6.00·10 ⁻⁶	normal	310·10 ⁻¹⁸	1.8·10 ⁻²¹ mol	0.0 %
R _{236/238X}	30.2070·10 ⁻⁶	62.0·10 ⁻⁶	normal	310·10 ⁻¹⁸	19·10 ⁻²⁴ mol	0.0 %
R _{238/238X}	1.0					
R _{blank} ¹	77.00	1.00	normal	-4.1·10 ⁻¹⁸	-4.1·10 ⁻¹⁸ mol	100.0 %
V	841.8·10 ⁻⁹ L	33.2·10 ⁻⁹ L		0.0	0.0 mol	0.0 %
w _{233Y}	9.01300·10 ⁻¹² mol/g	6.00·10 ⁻¹⁵ mol/g	normal	0.0	0.0 mol	0.0 %
X _{233Y}	0.999633200	700·10 ⁻⁶	normal	0.0	0.0 mol	0.0 %
ρ _y	1.03315 g/mL	2.04·10 ⁻³ g/mL		0.0	0.0 mol	0.0 %
n _{blank} ¹	315.62·10 ⁻¹⁸ mol	4.10·10 ⁻¹⁸ mol				
δn_{ip}: Uncertainty contribution due to spike addition						
Quantity	Value	Standard Uncertainty	Distribution	Sensitivity Coefficient	Uncertainty Contribution	Index
C ₂	1000.0 mL/L					
N _a	3.0					
V	841.8·10 ⁻⁹ L	33.2·10 ⁻⁹ L		1.2·10 ⁶	0.039	99.7 %
w _{233Y}	9.01300·10 ⁻¹² mol/g	6.00·10 ⁻¹⁵ mol/g	normal	110·10 ⁹	670·10 ⁻⁶	0.0 %
X _{233Y}	0.999633200	700·10 ⁻⁶	normal	-1.0	-700·10 ⁻⁹	0.0 %
ρ _y	1.03315 g/mL	2.04·10 ⁻³ g/mL		0.97	2.0·10 ⁻³	0.2 %
δn _{ip}	1.0000	0.0395				
Intermediate-precision uncertainty, including the factors from the pipetted volume, spike concentration and spike density.						
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ID-TIMS results for certification of U amount per particle in IRMM-2329P

n_{char} combined: Average uranium amount per particle with combined uncertainty for characterisation study

Quantity	Value	Standard Uncertainty	Distribution	Sensitivity Coefficient	Uncertainty Contribution	Index
C ₁	1.0·10 ¹⁵ fmol/mol					
C ₂	1000.0 mL/L					
N	10.0					
N _a	3.0					
R _{233/233Y}	1.0					
R _{234/233Y}	352.400·10 ⁻⁶	700·10 ⁻⁹	normal	-15	-11·10 ⁻⁶ fmol	0.0 %
R _{235/233Y}	4.1240·10 ⁻⁶	14.5·10 ⁻⁹	normal	-15	-220·10 ⁻⁶ fmol	0.0 %
R _{236/233Y}	43.400·10 ⁻⁹	700·10 ⁻¹²	normal	-15	-11·10 ⁻⁹ fmol	0.0 %
R _{238/233Y}	10.430·10 ⁻⁶	105·10 ⁻⁹	normal	-15	-1.6·10 ⁻⁶ fmol	0.0 %
R _{238/238Y}	1.0					
R _{233/238X}	0.0					
R _{234/238X}	340.8250·10 ⁻⁶	93.0·10 ⁻⁹	normal	15	1.4·10 ⁻⁶ fmol	0.0 %
R _{235/238X}	0.03390200	6.00·10 ⁻⁶	normal	15	87·10 ⁻⁶ fmol	0.0 %
R _{238/238X}	30.2070·10 ⁻⁶	62.0·10 ⁻⁹	normal	15	900·10 ⁻⁹ fmol	0.0 %
R _{238/238X}	1.0					
R _{blank1}	77.00	1.00	normal	160·10 ⁻⁶	160·10 ⁻⁶ fmol	0.0 %
R _{blank2}	57.00	2.00	normal	97·10 ⁻⁶	190·10 ⁻⁶ fmol	0.0 %
R _{blank3}	28.00	2.00	normal	not valid!	3.0·10 ⁻³ fmol	0.0 %
R _{T1T016F1}	0.154490	245·10 ⁻⁶	normal	-3.3	-810·10 ⁻⁶ fmol	0.0 %
R _{T1T016F2}	0.138370	165·10 ⁻⁶	normal	-4.1	-680·10 ⁻⁶ fmol	0.0 %
R _{T1T016F4}	0.134020	180·10 ⁻⁶	normal	-4.4	-790·10 ⁻⁶ fmol	0.0 %
R _{T1T016F5}	0.173750	370·10 ⁻⁶	normal	-2.6	-960·10 ⁻⁶ fmol	0.0 %
R _{T1T051F2}	0.163490	205·10 ⁻⁶	normal	-2.9	-600·10 ⁻⁶ fmol	0.0 %
R _{T1T051F3}	0.159500	305·10 ⁻⁶	normal	-3.1	-940·10 ⁻⁶ fmol	0.0 %
R _{T1T051F4}	0.156690	345·10 ⁻⁶	normal	-3.2	-1.1·10 ⁻³ fmol	0.0 %
R _{T1T051F5}	0.170270	445·10 ⁻⁶	normal	-2.7	-1.2·10 ⁻³ fmol	0.0 %
R _{T1T078F1}	0.145740	115·10 ⁻⁶	normal	-3.7	-420·10 ⁻⁶ fmol	0.0 %
R _{T1T078F2}	0.143520	240·10 ⁻⁶	normal	-3.8	-910·10 ⁻⁶ fmol	0.0 %
R _{T1T078F4}	0.149320	175·10 ⁻⁶	normal	-3.5	-620·10 ⁻⁶ fmol	0.0 %
R _{T1T078F5}	0.144740	230·10 ⁻⁶	normal	-3.7	-860·10 ⁻⁶ fmol	0.0 %
R _{T2T020F1}	0.176000	600·10 ⁻⁶	normal	-2.5	-1.5·10 ⁻³ fmol	0.0 %
R _{T2T066F4}	0.14620	1.10·10 ⁻³	normal	-3.7	-4.0·10 ⁻³ fmol	0.0 %
R _{T2T102F1}	0.16120	1.10·10 ⁻³	normal	-3.0	-3.3·10 ⁻³ fmol	0.0 %
R _{T2T102F3}	0.170400	440·10 ⁻⁶	normal	-2.7	-1.2·10 ⁻³ fmol	0.0 %
R _{T3T020F1}	0.176240	430·10 ⁻⁶	normal	-2.5	-1.1·10 ⁻³ fmol	0.0 %
R _{T3T020F2}	0.171780	375·10 ⁻⁶	normal	-2.7	-1.0·10 ⁻³ fmol	0.0 %

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ID-TIMS results for certification of U amount per particle in IRMM-2329P						
Quantity	Value	Standard Uncertainty	Distribution	Sensitivity Coefficient	Uncertainty Contribution	Index
R _{T3T020F3}	0.199240	170·10 ⁻⁶	normal	-2.0	-340·10 ⁻⁶ fmol	0.0 %
R _{T3T020F4}	0.191010	405·10 ⁻⁶	normal	-2.2	-870·10 ⁻⁶ fmol	0.0 %
R _{T3T020F5}	0.184300	550·10 ⁻⁶	normal	-2.3	-1.3·10 ⁻³ fmol	0.0 %
R _{T3T069F1}	0.168010	400·10 ⁻⁶	normal	-2.8	-1.1·10 ⁻³ fmol	0.0 %
R _{T3T069F2}	0.160500	270·10 ⁻⁶	normal	-3.0	-820·10 ⁻⁶ fmol	0.0 %
R _{T3T069F3}	0.153170	290·10 ⁻⁶	normal	-3.3	-970·10 ⁻⁶ fmol	0.0 %
R _{T3T069F4}	0.159470	370·10 ⁻⁶	normal	-3.1	-1.1·10 ⁻³ fmol	0.0 %
R _{T3T069F5}	0.161860	295·10 ⁻⁶	normal	-3.0	-880·10 ⁻⁶ fmol	0.0 %
R _{T3T102F1}	0.155960	400·10 ⁻⁶	normal	-3.2	-1.3·10 ⁻³ fmol	0.0 %
R _{T3T102F2}	0.162310	250·10 ⁻⁶	normal	-3.0	-740·10 ⁻⁶ fmol	0.0 %
R _{T3T102F3}	0.164830	300·10 ⁻⁶	normal	-2.9	-870·10 ⁻⁶ fmol	0.0 %
R _{T3T102F4}	0.164010	260·10 ⁻⁶	normal	-2.9	-760·10 ⁻⁶ fmol	0.0 %
R _{T3T102F5}	0.168280	265·10 ⁻⁶	normal	-2.8	-730·10 ⁻⁶ fmol	0.0 %
V	841.8·10 ⁻⁶ L	33.2·10 ⁻⁹ L		18·10 ⁶	0.59 fmol	99.7 %
w _{233Y}	9.01300·10 ⁻¹² mol/g	6.00·10 ⁻¹⁵ mol/g	normal	1.7·10 ¹²	0.010 fmol	0.0 %
X _{233Y}	0.999633200	700·10 ⁻⁹	normal	-15	-11·10 ⁻⁶ fmol	0.0 %
ρ _y	1.03315 g/mL	2.04·10 ⁻³ g/mL		15	0.030 fmol	0.2 %
n _{char,combined}	15.052 fmol	0.595 fmol				

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ID-TIMS results for certification of U amount per particle in IRMM-2329P						
m_{char} combined: Average uranium mass per particle with combined uncertainty for characterisation study						
Quantity	Value	Standard Uncertainty	Distribution	Sensitivity Coefficient	Uncertainty Contribution	Index
C ₁	1.0·10 ⁻¹⁵ fmol/mol					
C ₂	1000.0 mL/L					
C ₃	1.0·10 ⁻³ pg/fg					
M ₂₃₃	233.03963440 g/mol	2.40·10 ⁻⁶ g/mol	normal	0.0	0.0 pg	0.0 %
M ₂₃₄	234.04095040 g/mol	1.20·10 ⁻⁶ g/mol	normal	5.0·10 ⁻⁶	6.0·10 ⁻¹² pg	0.0 %
M ₂₃₅	235.04392820 g/mol	1.20·10 ⁻⁶ g/mol	normal	490·10 ⁻⁶	590·10 ⁻¹² pg	0.0 %
M ₂₃₆	236.04556620 g/mol	1.20·10 ⁻⁶ g/mol	normal	440·10 ⁻⁶	530·10 ⁻¹⁵ pg	0.0 %
M ₂₃₈	238.05078700 g/mol	1.60·10 ⁻⁶ g/mol	normal	0.015	23·10 ⁻⁹ pg	0.0 %
N	10.0					
N _s	3.0					
R _{233/233Y}	1.0					
R _{234/233Y}	352.400·10 ⁻⁶	700·10 ⁻⁹	normal	-3.6	-2.5·10 ⁻⁶ pg	0.0 %
R _{235/233Y}	4.1240·10 ⁻⁶	14.5·10 ⁻⁹	normal	-3.6	-52·10 ⁻⁹ pg	0.0 %
R _{236/233Y}	43.400·10 ⁻⁹	700·10 ⁻¹²	normal	-3.6	-2.5·10 ⁻⁹ pg	0.0 %
R _{238/233Y}	10.430·10 ⁻⁶	105·10 ⁻⁹	normal	-3.6	-380·10 ⁻⁹ pg	0.0 %
R _{238/238Y}	1.0					
R _{233/238X}	0.0					
R _{234/238X}	340.8250·10 ⁻⁶	93.0·10 ⁻⁹	normal	3.4	320·10 ⁻⁹ pg	0.0 %
R _{235/238X}	0.03390200	6.00·10 ⁻⁶	normal	3.4	21·10 ⁻⁶ pg	0.0 %
R _{236/238X}	30.2070·10 ⁻⁶	62.0·10 ⁻⁹	normal	3.4	210·10 ⁻⁹ pg	0.0 %
R _{238/238X}	1.0					
R _{blank1}	77.00	1.00	normal	38·10 ⁻⁶	38·10 ⁻⁶ pg	0.0 %
R _{blank2}	57.00	2.00	normal	23·10 ⁻⁶	46·10 ⁻⁶ pg	0.0 %
R _{blank3}	28.00	2.00	normal	not valid!	720·10 ⁻⁶ pg	0.0 %
R _{T1T016F1}	0.154490	245·10 ⁻⁶	normal	-0.78	-190·10 ⁻⁶ pg	0.0 %
R _{T1T016F2}	0.138370	165·10 ⁻⁶	normal	-0.98	-160·10 ⁻⁶ pg	0.0 %
R _{T1T016F4}	0.134020	180·10 ⁻⁶	normal	-1.0	-190·10 ⁻⁶ pg	0.0 %
R _{T1T016F5}	0.173750	370·10 ⁻⁶	normal	-0.62	-230·10 ⁻⁶ pg	0.0 %
R _{T1T051F2}	0.163490	205·10 ⁻⁶	normal	-0.70	-140·10 ⁻⁶ pg	0.0 %
R _{T1T051F3}	0.159500	305·10 ⁻⁶	normal	-0.73	-220·10 ⁻⁶ pg	0.0 %
R _{T1T051F4}	0.156690	345·10 ⁻⁶	normal	-0.76	-260·10 ⁻⁶ pg	0.0 %
R _{T1T051F5}	0.170270	445·10 ⁻⁶	normal	-0.64	-290·10 ⁻⁶ pg	0.0 %
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ID-TIMS results for certification of U amount per particle in IRMM-2329P						
Quantity	Value	Standard Uncertainty	Distribution	Sensitivity Coefficient	Uncertainty Contribution	Index
R _{T1T078F1}	0.145740	115·10 ⁻⁶	normal	-0.88	-100·10 ⁻⁶ pg	0.0 %
R _{T1T078F2}	0.143520	240·10 ⁻⁶	normal	-0.91	-220·10 ⁻⁶ pg	0.0 %
R _{T1T078F4}	0.149320	175·10 ⁻⁶	normal	-0.84	-150·10 ⁻⁶ pg	0.0 %
R _{T1T078F5}	0.144740	230·10 ⁻⁶	normal	-0.89	-200·10 ⁻⁶ pg	0.0 %
R _{T2T020F1}	0.176000	600·10 ⁻⁶	normal	-0.60	-360·10 ⁻⁶ pg	0.0 %
R _{T2T089F4}	0.14620	1.10·10 ⁻³	normal	-0.87	-960·10 ⁻⁶ pg	0.0 %
R _{T2T102F1}	0.16120	1.10·10 ⁻³	normal	-0.72	-790·10 ⁻⁶ pg	0.0 %
R _{T2T102F3}	0.170400	440·10 ⁻⁶	normal	-0.64	-280·10 ⁻⁶ pg	0.0 %
R _{T3T020F1}	0.176240	430·10 ⁻⁶	normal	-0.60	-260·10 ⁻⁶ pg	0.0 %
R _{T3T020F2}	0.171780	375·10 ⁻⁶	normal	-0.63	-240·10 ⁻⁶ pg	0.0 %
R _{T3T020F3}	0.199240	170·10 ⁻⁶	normal	-0.47	-80·10 ⁻⁶ pg	0.0 %
R _{T3T020F4}	0.191010	405·10 ⁻⁶	normal	-0.51	-210·10 ⁻⁶ pg	0.0 %
R _{T3T020F5}	0.184300	550·10 ⁻⁶	normal	-0.55	-300·10 ⁻⁶ pg	0.0 %
R _{T3T089F1}	0.168010	400·10 ⁻⁶	normal	-0.66	-260·10 ⁻⁶ pg	0.0 %
R _{T3T089F2}	0.160500	270·10 ⁻⁶	normal	-0.72	-200·10 ⁻⁶ pg	0.0 %
R _{T3T089F3}	0.153170	290·10 ⁻⁶	normal	-0.80	-230·10 ⁻⁶ pg	0.0 %
R _{T3T089F4}	0.159470	370·10 ⁻⁶	normal	-0.73	-270·10 ⁻⁶ pg	0.0 %
R _{T3T089F5}	0.161860	295·10 ⁻⁶	normal	-0.71	-210·10 ⁻⁶ pg	0.0 %
R _{T3T102F1}	0.155960	400·10 ⁻⁶	normal	-0.77	-310·10 ⁻⁶ pg	0.0 %
R _{T3T102F2}	0.162310	250·10 ⁻⁶	normal	-0.71	-180·10 ⁻⁶ pg	0.0 %
R _{T3T102F3}	0.164830	300·10 ⁻⁶	normal	-0.69	-210·10 ⁻⁶ pg	0.0 %
R _{T3T102F4}	0.164010	260·10 ⁻⁶	normal	-0.69	-180·10 ⁻⁶ pg	0.0 %
R _{T3T102F5}	0.168280	265·10 ⁻⁶	normal	-0.66	-170·10 ⁻⁶ pg	0.0 %
V	841.8·10 ⁻⁹ L	33.2·10 ⁻⁹ L		4.3·10 ⁶	0.14 pg	99.7 %
w _{233Y}	9.01300·10 ⁻¹² mol/g	6.00·10 ⁻¹⁵ mol/g	normal	400·10 ⁹	2.4·10 ⁻³ pg	0.0 %
X _{233Y}	0.999633200	700·10 ⁻⁹	normal	-3.6	-2.5·10 ⁻⁶ pg	0.0 %
ρ _y	1.03315 g/mL	2.04·10 ⁻³ g/mL		3.5	7.1·10 ⁻³ pg	0.2 %
m _{char,CO} mbined	3.582 pg	0.142 pg				

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ID-TIMS results for certification of U amount per particle in IRMM-2329P

n_{CRM} : Uranium amount per particle of certified particles

Quantity	Value	Standard Uncertainty	Distribution	Sensitivity Coefficient	Uncertainty Contribution	Index
C ₁	1.0·10 ¹⁵ fmol/mol					
C ₂	1000.0 mL/L					
N	10.0					
N _a	3.0					
R _{233/233Y}	1.0					
R _{234/233Y}	352.400·10 ⁻⁶	700·10 ⁻⁹	normal	-15	-11·10 ⁻⁶ fmol	0.0 %
R _{235/233Y}	4.1240·10 ⁻⁶	14.5·10 ⁻⁹	normal	-15	-220·10 ⁻⁹ fmol	0.0 %
R _{236/233Y}	43.400·10 ⁻⁹	700·10 ⁻¹²	normal	-15	-11·10 ⁻⁹ fmol	0.0 %
R _{238/233Y}	10.430·10 ⁻⁶	105·10 ⁻⁹	normal	-15	-1.6·10 ⁻⁶ fmol	0.0 %
R _{238/238Y}	1.0					
R _{233/238X}	0.0					
R _{234/238X}	340.8250·10 ⁻⁶	93.0·10 ⁻⁹	normal	15	1.4·10 ⁻⁶ fmol	0.0 %
R _{235/238X}	0.03390200	6.00·10 ⁻⁶	normal	15	87·10 ⁻⁵ fmol	0.0 %
R _{236/238X}	30.2070·10 ⁻⁶	62.0·10 ⁻⁹	normal	15	900·10 ⁻⁹ fmol	0.0 %
R _{238/238X}	1.0					
R _{blank1}	77.00	1.00	normal	160·10 ⁻⁶	160·10 ⁻⁶ fmol	0.0 %
R _{blank2}	57.00	2.00	normal	97·10 ⁻⁶	190·10 ⁻⁶ fmol	0.0 %
R _{blank3}	28.00	2.00	normal	not valid!	3.0·10 ⁻³ fmol	0.0 %
R _{T1T018F1}	0.154490	245·10 ⁻⁶	normal	-3.3	-810·10 ⁻⁶ fmol	0.0 %
R _{T1T018F2}	0.138370	165·10 ⁻⁶	normal	-4.1	-680·10 ⁻⁶ fmol	0.0 %
R _{T1T018F4}	0.134020	180·10 ⁻⁶	normal	-4.4	-790·10 ⁻⁶ fmol	0.0 %
R _{T1T018F5}	0.173750	370·10 ⁻⁶	normal	-2.6	-960·10 ⁻⁶ fmol	0.0 %
R _{T1T051F2}	0.163490	205·10 ⁻⁶	normal	-2.9	-600·10 ⁻⁶ fmol	0.0 %
R _{T1T051F3}	0.159500	305·10 ⁻⁶	normal	-3.1	-940·10 ⁻⁶ fmol	0.0 %
R _{T1T051F4}	0.156690	345·10 ⁻⁶	normal	-3.2	-1.1·10 ⁻³ fmol	0.0 %
R _{T1T051F5}	0.170270	445·10 ⁻⁶	normal	-2.7	-1.2·10 ⁻³ fmol	0.0 %
R _{T1T078F1}	0.145740	115·10 ⁻⁶	normal	-3.7	-420·10 ⁻⁶ fmol	0.0 %
R _{T1T078F2}	0.143520	240·10 ⁻⁶	normal	-3.8	-910·10 ⁻⁶ fmol	0.0 %
R _{T1T078F4}	0.149320	175·10 ⁻⁶	normal	-3.5	-620·10 ⁻⁶ fmol	0.0 %
R _{T1T078F5}	0.144740	230·10 ⁻⁶	normal	-3.7	-860·10 ⁻⁶ fmol	0.0 %
R _{T2T020F1}	0.176000	600·10 ⁻⁶	normal	-2.5	-1.5·10 ⁻³ fmol	0.0 %
R _{T2T088F4}	0.14620	1.10·10 ⁻³	normal	-3.7	-4.0·10 ⁻³ fmol	0.0 %
R _{T2T102F1}	0.16120	1.10·10 ⁻³	normal	-3.0	-3.3·10 ⁻³ fmol	0.0 %
R _{T2T102F3}	0.170400	440·10 ⁻⁶	normal	-2.7	-1.2·10 ⁻³ fmol	0.0 %
R _{T3T020F1}	0.176240	430·10 ⁻⁶	normal	-2.5	-1.1·10 ⁻³ fmol	0.0 %
R _{T3T020F2}	0.171780	375·10 ⁻⁶	normal	-2.7	-1.0·10 ⁻³ fmol	0.0 %

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ID-TIMS results for certification of U amount per particle in
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Quantity	Value	Standard Uncertainty	Distribution	Sensitivity Coefficient	Uncertainty Contribution	Index
R _{T3T020F3}	0.199240	170·10 ⁻⁶	normal	-2.0	-340·10 ⁻⁶ fmol	0.0 %
R _{T3T020F4}	0.191010	405·10 ⁻⁶	normal	-2.2	-870·10 ⁻⁶ fmol	0.0 %
R _{T3T020F5}	0.184300	550·10 ⁻⁶	normal	-2.3	-1.3·10 ⁻³ fmol	0.0 %
R _{T3T089F1}	0.168010	400·10 ⁻⁶	normal	-2.8	-1.1·10 ⁻³ fmol	0.0 %
R _{T3T089F2}	0.160500	270·10 ⁻⁶	normal	-3.0	-820·10 ⁻⁶ fmol	0.0 %
R _{T3T089F3}	0.153170	290·10 ⁻⁶	normal	-3.3	-970·10 ⁻⁶ fmol	0.0 %
R _{T3T089F4}	0.159470	370·10 ⁻⁶	normal	-3.1	-1.1·10 ⁻³ fmol	0.0 %
R _{T3T089F5}	0.161860	295·10 ⁻⁶	normal	-3.0	-880·10 ⁻⁶ fmol	0.0 %
R _{T3T102F1}	0.155960	400·10 ⁻⁶	normal	-3.2	-1.3·10 ⁻³ fmol	0.0 %
R _{T3T102F2}	0.162310	250·10 ⁻⁶	normal	-3.0	-740·10 ⁻⁶ fmol	0.0 %
R _{T3T102F3}	0.164830	300·10 ⁻⁶	normal	-2.9	-870·10 ⁻⁶ fmol	0.0 %
R _{T3T102F4}	0.164010	260·10 ⁻⁶	normal	-2.9	-760·10 ⁻⁶ fmol	0.0 %
R _{T3T102F5}	0.168280	265·10 ⁻⁶	normal	-2.8	-730·10 ⁻⁶ fmol	0.0 %
V	841.8·10 ⁻⁹ L	33.2·10 ⁻⁹ L		18·10 ⁶	0.59 fmol	20.5 %
w _{233Y}	9.01300·10 ⁻¹² mol/g	6.00·10 ⁻¹³ mol/g	normal	1.7·10 ¹²	0.010 fmol	0.0 %
X _{233Y}	0.999633200	700·10 ⁻⁹	normal	-15	-11·10 ⁻⁶ fmol	0.0 %
ρ _y	1.03315 g/mL	2.04·10 ⁻³ g/mL		15	0.030 fmol	0.0 %
δx _{bb}	1.0000	0.0550		15	0.83 fmol	39.7 %
δx _{swb}	1.0000	0.0550		15	0.83 fmol	39.7 %
n _{CRM}	15.05 fmol	1.31 fmol				

Uranium amount per particle, the main certified value

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ID-TIMS results for certification of U amount per particle in
IRMM-2329P

m_{CRM}: Uranium mass per particle of certified particles

Quantity	Value	Standard Uncertainty	Distribution	Sensitivity Coefficient	Uncertainty Contribution	Index
C ₁	1.0·10 ¹⁵ fmol/mol					
C ₂	1000.0 mL/L					
C ₃	1.0·10 ⁻³ pg/fg					
M ₂₃₃	233.03963440 g/mol	2.40·10 ⁻⁶ g/mol	normal	0.0	0.0 pg	0.0 %
M ₂₃₄	234.04095040 g/mol	1.20·10 ⁻⁶ g/mol	normal	5.0·10 ⁻⁶	6.0·10 ⁻¹² pg	0.0 %
M ₂₃₅	235.04392820 g/mol	1.20·10 ⁻⁶ g/mol	normal	490·10 ⁻⁶	590·10 ⁻¹² pg	0.0 %
M ₂₃₆	236.04556620 g/mol	1.20·10 ⁻⁶ g/mol	normal	440·10 ⁻⁹	530·10 ⁻¹⁵ pg	0.0 %
M ₂₃₈	238.05078700 g/mol	1.60·10 ⁻⁶ g/mol	normal	0.015	23·10 ⁻⁹ pg	0.0 %
N	10.0					
N _a	3.0					
R _{233/233} ^Y	1.0					
R _{234/233} ^Y	352.400·10 ⁻⁶	700·10 ⁻⁹	normal	-3.6	-2.5·10 ⁻⁶ pg	0.0 %
R _{235/233} ^Y	4.1240·10 ⁻⁶	14.5·10 ⁻⁹	normal	-3.6	-52·10 ⁻⁹ pg	0.0 %
R _{236/233} ^Y	43.400·10 ⁻⁹	700·10 ⁻¹²	normal	-3.6	-2.5·10 ⁻⁹ pg	0.0 %
R _{238/233} ^Y	10.430·10 ⁻⁶	105·10 ⁻⁹	normal	-3.6	-380·10 ⁻⁹ pg	0.0 %
R _{238/238} ^Y	1.0					
R _{233/238} ^X	0.0					
R _{234/238} ^X	340.8250·10 ⁻⁶	93.0·10 ⁻⁹	normal	3.4	320·10 ⁻⁹ pg	0.0 %
R _{235/238} ^X	0.03390200	6.00·10 ⁻⁹	normal	3.4	21·10 ⁻⁹ pg	0.0 %
R _{236/238} ^X	30.2070·10 ⁻⁶	62.0·10 ⁻⁹	normal	3.4	210·10 ⁻⁹ pg	0.0 %
R _{238/238} ^X	1.0					
R _{blank1}	77.00	1.00	normal	38·10 ⁻⁶	38·10 ⁻⁶ pg	0.0 %
R _{blank2}	57.00	2.00	normal	23·10 ⁻⁶	46·10 ⁻⁶ pg	0.0 %
R _{blank3}	28.00	2.00	normal	not valid!	720·10 ⁻⁶ pg	0.0 %
R _{T1T016F1}	0.154490	245·10 ⁻⁶	normal	-0.78	-190·10 ⁻⁶ pg	0.0 %
R _{T1T016F2}	0.138370	165·10 ⁻⁶	normal	-0.98	-160·10 ⁻⁶ pg	0.0 %
R _{T1T016F4}	0.134020	180·10 ⁻⁶	normal	-1.0	-190·10 ⁻⁶ pg	0.0 %
R _{T1T016F5}	0.173750	370·10 ⁻⁶	normal	-0.62	-230·10 ⁻⁶ pg	0.0 %
R _{T1T051F2}	0.163490	205·10 ⁻⁶	normal	-0.70	-140·10 ⁻⁶ pg	0.0 %
R _{T1T051F3}	0.159500	305·10 ⁻⁶	normal	-0.73	-220·10 ⁻⁶ pg	0.0 %
R _{T1T051F4}	0.156690	345·10 ⁻⁶	normal	-0.76	-260·10 ⁻⁶ pg	0.0 %
R _{T1T051F5}	0.170270	445·10 ⁻⁶	normal	-0.64	-290·10 ⁻⁶ pg	0.0 %
R _{T1T078F1}	0.145740	115·10 ⁻⁶	normal	-0.88	-100·10 ⁻⁶ pg	0.0 %

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ID-TIMS results for certification of U amount per particle in IRMM-2329P						
Quantity	Value	Standard Uncertainty	Distribution	Sensitivity Coefficient	Uncertainty Contribution	Index
R _{T1T078F2}	0.143520	240·10 ⁻⁶	normal	-0.91	-220·10 ⁻⁶ pg	0.0 %
R _{T1T078F4}	0.149320	175·10 ⁻⁶	normal	-0.84	-150·10 ⁻⁶ pg	0.0 %
R _{T1T078F5}	0.144740	230·10 ⁻⁶	normal	-0.89	-200·10 ⁻⁶ pg	0.0 %
R _{T2T020F1}	0.176000	600·10 ⁻⁶	normal	-0.60	-360·10 ⁻⁶ pg	0.0 %
R _{T2T069F4}	0.14620	1.10·10 ⁻³	normal	-0.87	-960·10 ⁻⁶ pg	0.0 %
R _{T2T102F1}	0.16120	1.10·10 ⁻³	normal	-0.72	-790·10 ⁻⁶ pg	0.0 %
R _{T2T102F3}	0.170400	440·10 ⁻⁶	normal	-0.64	-280·10 ⁻⁶ pg	0.0 %
R _{T3T020F1}	0.176240	430·10 ⁻⁶	normal	-0.60	-260·10 ⁻⁶ pg	0.0 %
R _{T3T020F2}	0.171780	375·10 ⁻⁶	normal	-0.63	-240·10 ⁻⁶ pg	0.0 %
R _{T3T020F3}	0.199240	170·10 ⁻⁶	normal	-0.47	-80·10 ⁻⁶ pg	0.0 %
R _{T3T020F4}	0.191010	405·10 ⁻⁶	normal	-0.51	-210·10 ⁻⁶ pg	0.0 %
R _{T3T020F5}	0.184300	550·10 ⁻⁶	normal	-0.55	-300·10 ⁻⁶ pg	0.0 %
R _{T3T069F1}	0.168010	400·10 ⁻⁶	normal	-0.66	-260·10 ⁻⁶ pg	0.0 %
R _{T3T069F2}	0.160500	270·10 ⁻⁶	normal	-0.72	-200·10 ⁻⁶ pg	0.0 %
R _{T3T069F3}	0.153170	290·10 ⁻⁶	normal	-0.80	-230·10 ⁻⁶ pg	0.0 %
R _{T3T069F4}	0.159470	370·10 ⁻⁶	normal	-0.73	-270·10 ⁻⁶ pg	0.0 %
R _{T3T069F5}	0.161860	295·10 ⁻⁶	normal	-0.71	-210·10 ⁻⁶ pg	0.0 %
R _{T3T102F1}	0.155960	400·10 ⁻⁶	normal	-0.77	-310·10 ⁻⁶ pg	0.0 %
R _{T3T102F2}	0.162310	250·10 ⁻⁶	normal	-0.71	-180·10 ⁻⁶ pg	0.0 %
R _{T3T102F3}	0.164830	300·10 ⁻⁶	normal	-0.69	-210·10 ⁻⁶ pg	0.0 %
R _{T3T102F4}	0.164010	260·10 ⁻⁶	normal	-0.69	-180·10 ⁻⁶ pg	0.0 %
R _{T3T102F5}	0.168280	265·10 ⁻⁶	normal	-0.66	-170·10 ⁻⁶ pg	0.0 %
V	841.8·10 ⁻⁹ L	33.2·10 ⁻⁹ L		4.3·10 ⁶	0.14 pg	20.5 %
w _{233Y}	9.01300·10 ⁻¹² mol/g	6.00·10 ⁻¹⁵ mol/g	normal	400·10 ⁶	2.4·10 ⁻³ pg	0.0 %
X _{233Y}	0.999633200	700·10 ⁻⁹	normal	-3.6	-2.5·10 ⁻⁶ pg	0.0 %
ρ _y	1.03315 g/mL	2.04·10 ⁻³ g/mL		3.5	7.1·10 ⁻³ pg	0.0 %
δx _{bb}	1.0000	0.0550		3.6	0.20 pg	39.7 %
δx _{wb}	1.0000	0.0550		3.6	0.20 pg	39.7 %
m _{CRM}	3.582 pg	0.313 pg				
Uranium mass per particles, derived from the uranium amount per particle and the molar mass (derived from certified isotopic composition)						
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ID-TIMS results for certification of U amount per particle in IRMM-2329P

N_{CRM}: Number of atoms per particle

Quantity	Value	Standard Uncertainty	Distribution	Sensitivity Coefficient	Uncertainty Contribution	Index
C ₁	1.0·10 ¹⁵ fmol/mol					
C ₂	1000.0 mL/L					
N	10.0					
N _a	3.0					
R _{233/233Y}	1.0					
R _{234/233Y}	352.400·10 ⁻⁶	700·10 ⁻⁹	normal	-9.1·10 ⁹	-6300	0.0 %
R _{235/233Y}	4.1240·10 ⁻⁶	14.5·10 ⁻⁹	normal	-9.1·10 ⁹	-130	0.0 %
R _{236/233Y}	43.400·10 ⁻⁹	700·10 ⁻¹²	normal	-9.1·10 ⁹	-6.3	0.0 %
R _{238/233Y}	10.430·10 ⁻⁶	105·10 ⁻⁹	normal	-9.1·10 ⁹	-950	0.0 %
R _{238/238Y}	1.0					
R _{233/238X}	0.0					
R _{234/238X}	340.8250·10 ⁻⁶	93.0·10 ⁻⁹	normal	8.8·10 ⁹	820	0.0 %
R _{235/238X}	0.03390200	6.00·10 ⁻⁶	normal	8.8·10 ⁹	53000	0.0 %
R _{236/238X}	30.2070·10 ⁻⁶	62.0·10 ⁻⁹	normal	8.8·10 ⁹	540	0.0 %
R _{238/238X}	1.0					
R _{blank1}	77.00	1.00	normal	96000	96000	0.0 %
R _{blank2}	57.00	2.00	normal	58000	120·10 ³	0.0 %
R _{blank3}	28.00	2.00	normal	not valid!	1.8·10 ⁶	0.0 %
R _{T1T016F1}	0.154490	245·10 ⁻⁶	normal	-2.0·10 ⁹	-490·10 ³	0.0 %
R _{T1T016F2}	0.138370	165·10 ⁻⁶	normal	-2.5·10 ⁹	-410·10 ³	0.0 %
R _{T1T016F4}	0.134020	180·10 ⁻⁶	normal	-2.6·10 ⁹	-470·10 ³	0.0 %
R _{T1T016F5}	0.173750	370·10 ⁻⁶	normal	-1.6·10 ⁹	-580·10 ³	0.0 %
R _{T1T051F2}	0.163490	205·10 ⁻⁶	normal	-1.8·10 ⁹	-360·10 ³	0.0 %
R _{T1T051F3}	0.159500	305·10 ⁻⁶	normal	-1.9·10 ⁹	-570·10 ³	0.0 %
R _{T1T051F4}	0.156690	345·10 ⁻⁶	normal	-1.9·10 ⁹	-660·10 ³	0.0 %
R _{T1T051F5}	0.170270	445·10 ⁻⁶	normal	-1.6·10 ⁹	-730·10 ³	0.0 %
R _{T1T078F1}	0.145740	115·10 ⁻⁶	normal	-2.2·10 ⁹	-260·10 ³	0.0 %
R _{T1T078F2}	0.143520	240·10 ⁻⁶	normal	-2.3·10 ⁹	-550·10 ³	0.0 %
R _{T1T078F4}	0.149320	175·10 ⁻⁶	normal	-2.1·10 ⁹	-370·10 ³	0.0 %
R _{T1T078F5}	0.144740	230·10 ⁻⁶	normal	-2.3·10 ⁹	-520·10 ³	0.0 %
R _{T2T020F1}	0.176000	600·10 ⁻⁶	normal	-1.5·10 ⁹	-920·10 ³	0.0 %
R _{T2T099F4}	0.14620	1.10·10 ⁻³	normal	-2.2·10 ⁹	-2.4·10 ⁶	0.0 %
R _{T2T102F1}	0.16120	1.10·10 ⁻³	normal	-1.8·10 ⁹	-2.0·10 ⁶	0.0 %
R _{T2T102F3}	0.170400	440·10 ⁻⁶	normal	-1.6·10 ⁹	-720·10 ³	0.0 %
R _{T3T020F1}	0.176240	430·10 ⁻⁶	normal	-1.5·10 ⁹	-650·10 ³	0.0 %
R _{T3T020F2}	0.171780	375·10 ⁻⁶	normal	-1.6·10 ⁹	-600·10 ³	0.0 %

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ID-TIMS results for certification of U amount per particle in IRMM-2329P

Quantity	Value	Standard Uncertainty	Distribution	Sensitivity Coefficient	Uncertainty Contribution	Index
R _{T3T020F3}	0.199240	170·10 ⁻⁶	normal	-1.2·10 ⁹	-200·10 ³	0.0 %
R _{T3T020F4}	0.191010	405·10 ⁻⁶	normal	-1.3·10 ⁹	-520·10 ³	0.0 %
R _{T3T020F5}	0.184300	550·10 ⁻⁶	normal	-1.4·10 ⁹	-770·10 ³	0.0 %
R _{T3T066F1}	0.168010	400·10 ⁻⁶	normal	-1.7·10 ⁹	-670·10 ³	0.0 %
R _{T3T066F2}	0.160500	270·10 ⁻⁶	normal	-1.8·10 ⁹	-500·10 ³	0.0 %
R _{T3T066F3}	0.153170	290·10 ⁻⁶	normal	-2.0·10 ⁹	-580·10 ³	0.0 %
R _{T3T066F4}	0.159470	370·10 ⁻⁶	normal	-1.9·10 ⁹	-690·10 ³	0.0 %
R _{T3T066F5}	0.161860	295·10 ⁻⁶	normal	-1.8·10 ⁹	-530·10 ³	0.0 %
R _{T3T102F1}	0.155960	400·10 ⁻⁶	normal	-1.9·10 ⁹	-780·10 ³	0.0 %
R _{T3T102F2}	0.162310	250·10 ⁻⁶	normal	-1.8·10 ⁹	-450·10 ³	0.0 %
R _{T3T102F3}	0.164830	300·10 ⁻⁶	normal	-1.7·10 ⁹	-520·10 ³	0.0 %
R _{T3T102F4}	0.164010	260·10 ⁻⁶	normal	-1.8·10 ⁹	-460·10 ³	0.0 %
R _{T3T102F5}	0.168280	265·10 ⁻⁶	normal	-1.7·10 ⁹	-440·10 ³	0.0 %
V	841.8·10 ⁻⁹ L	33.2·10 ⁻⁹ L		11·10 ¹⁵	360·10 ⁶	20.5 %
w _{233Y}	9.01300·10 ⁻¹² mol/g	6.00·10 ⁻¹⁵ mol/g	normal	1.0·10 ²¹	6.0·10 ⁶	0.0 %
X _{233Y}	0.999633200	700·10 ⁻⁹	normal	-9.1·10 ⁹	-6300	0.0 %
ρ _Y	1.03315 g/mL	2.04·10 ⁻³ g/mL		8.8·10 ⁹	18·10 ⁶	0.0 %
N _A	602.21408570·10 ²¹ mol ⁻¹	7.40·10 ¹⁵ mol ⁻¹	normal	15·10 ¹⁶	110	0.0 %
δx _{bb}	1.0000	0.0550		9.1·10 ⁹	500·10 ⁶	39.7 %
δx _{swb}	1.0000	0.0550		9.1·10 ⁹	500·10 ⁶	39.7 %
N _{CRM}	9.064·10 ⁹	791·10 ⁶				

Amount of uranium atoms per particle, derived from the uranium amount per particle and Avogadro's constant.

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ID-TIMS results for certification of U amount per particle in IRMM-2329P

Results:

Quantity	Value	Expanded Uncertainty	Coverage factor	Coverage
m _{T1T016F1}	15.712 fmol	0.050 fmol	2.00	95% (t-table 95.45%)
m _{T1T016F2}	17.546 fmol	0.042 fmol	2.00	95% (t-table 95.45%)
m _{T1T016F4}	18.117 fmol	0.049 fmol	2.00	95% (t-table 95.45%)
m _{T1T016F5}	13.967 fmol	0.060 fmol	2.00	95% (t-table 95.45%)
m _{T1T051F2}	14.845 fmol	0.037 fmol	2.00	95% (t-table 95.45%)
m _{T1T051F3}	15.218 fmol	0.058 fmol	2.00	95% (t-table 95.45%)
m _{T1T051F4}	15.491 fmol	0.068 fmol	2.00	95% (t-table 95.45%)
m _{T1T051F5}	14.253 fmol	0.075 fmol	2.00	95% (t-table 95.45%)
m _{T1T078F1}	16.657 fmol	0.026 fmol	2.00	95% (t-table 95.45%)
m _{T1T078F2}	16.915 fmol	0.057 fmol	2.00	95% (t-table 95.45%)
m _{T1T078F4}	16.257 fmol	0.038 fmol	2.00	95% (t-table 95.45%)
m _{T1T078F5}	16.773 fmol	0.053 fmol	2.00	95% (t-table 95.45%)
m _{T2T020F1}	13.777 fmol	0.094 fmol	2.00	95% (t-table 95.45%)
m _{T2T068F4}	16.59 fmol	0.25 fmol	2.00	95% (t-table 95.45%)
m _{T2T102F1}	15.05 fmol	0.21 fmol	2.00	95% (t-table 95.45%)
m _{T2T102F3}	14.231 fmol	0.074 fmol	2.00	95% (t-table 95.45%)
m _{T3T020F1}	13.714 fmol	0.068 fmol	2.00	95% (t-table 95.45%)
m _{T3T020F2}	14.072 fmol	0.063 fmol	2.00	95% (t-table 95.45%)
m _{T3T020F3}	12.121 fmol	0.024 fmol	2.00	95% (t-table 95.45%)
m _{T3T020F4}	12.647 fmol	0.055 fmol	2.00	95% (t-table 95.45%)
m _{T3T020F5}	13.110 fmol	0.080 fmol	2.00	95% (t-table 95.45%)
m _{T3T069F1}	14.390 fmol	0.070 fmol	2.00	95% (t-table 95.45%)
m _{T3T069F2}	15.067 fmol	0.052 fmol	2.00	95% (t-table 95.45%)
m _{T3T069F3}	15.792 fmol	0.061 fmol	2.00	95% (t-table 95.45%)
m _{T3T069F4}	15.165 fmol	0.072 fmol	2.00	95% (t-table 95.45%)
m _{T3T069F5}	14.940 fmol	0.056 fmol	2.00	95% (t-table 95.45%)
m _{T3T102F1}	15.508 fmol	0.081 fmol	2.00	95% (t-table 95.45%)
m _{T3T102F2}	14.898 fmol	0.048 fmol	2.00	95% (t-table 95.45%)
m _{T3T102F3}	14.669 fmol	0.055 fmol	2.00	95% (t-table 95.45%)
m _{T3T102F4}	14.743 fmol	0.049 fmol	2.00	95% (t-table 95.45%)
m _{T3T102F5}	14.367 fmol	0.047 fmol	2.00	95% (t-table 95.45%)
n _{char}	15.0517 fmol	0.053 % (relative)	1.00	manual
n _{blank1}	315.6·10 ⁻¹⁸ mol	8.2·10 ⁻¹⁸ mol	2.00	95% (t-table 95.45%)
n _{blank2}	426·10 ⁻¹⁸ mol	30·10 ⁻¹⁸ mol	2.00	95% (t-table 95.45%)
n _{blank3}	870·10 ⁻¹⁸ mol	120·10 ⁻¹⁸ mol	2.00	95% (t-table 95.45%)
n _{char,combined}	15.1 fmol	1.2 fmol	2.00	95% (normal)

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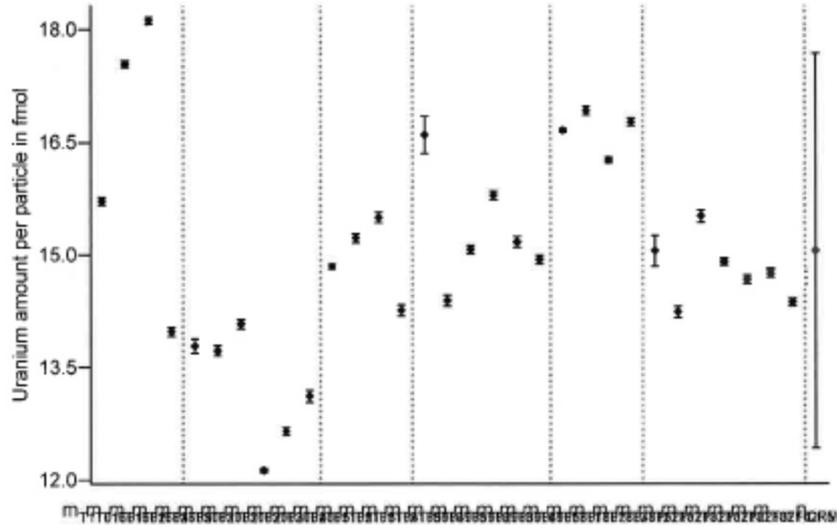
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ID-TIMS results for certification of U amount per particle in IRMM-2329P

Quantity	Value	Expanded Uncertainty	Coverage factor	Coverage
$m_{char,combined}$	3.58 pg	0.28 pg	2.00	95% (normal)
n_{CRM}	15.1 fmol	2.6 fmol	2.00	95% (normal)
m_{CRM}	3.58 pg	0.63 pg	2.00	95% (normal)
N_{CRM}	$9.1 \cdot 10^9$	$1.6 \cdot 10^9$	2.00	95% (normal)

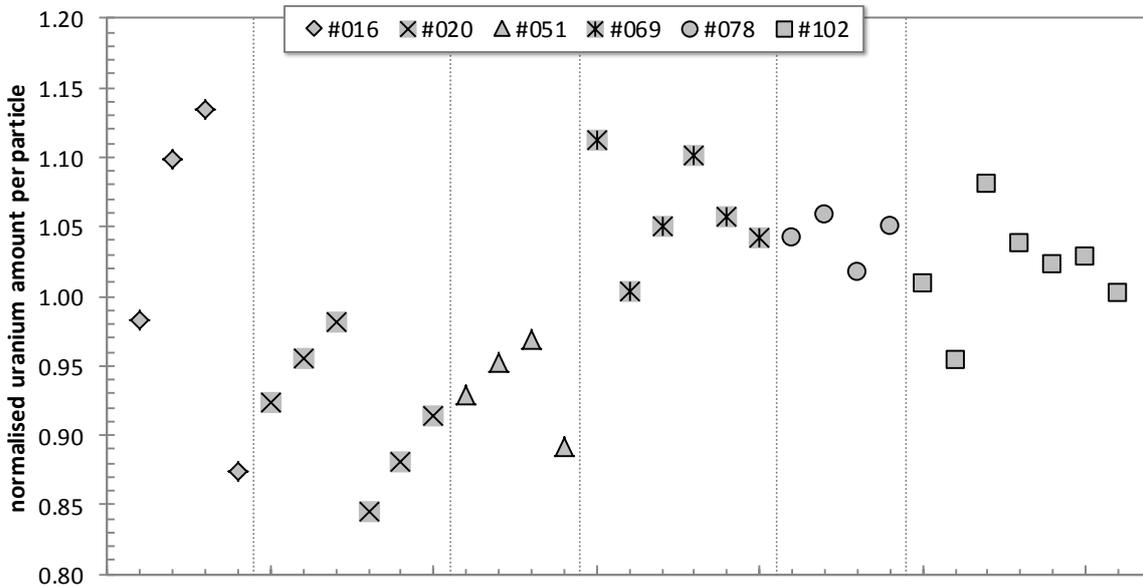


Annex 10. Homogeneity results

	#016	#020	#051	#069	#078	#102
1	0.983	0.924	REJECTED	REJECTED	1.042	1.009
2	1.098	REJECTED	0.929	REJECTED	1.059	REJECTED
3	REJECTED	REJECTED	0.952	REJECTED	REJECTED	0.954
4	1.134	REJECTED	0.969	1.113	1.017	REJECTED
5	0.874	REJECTED	0.892	REJECTED	1.050	REJECTED
6	N.A.	0.956	N.A.	1.003	N.A.	1.081
7	N.A.	0.981	N.A.	1.050	N.A.	1.038
8	N.A.	0.845	N.A.	1.101	N.A.	1.022
9	N.A.	0.882	N.A.	1.057	N.A.	1.028
10	N.A.	0.914	N.A.	1.041	N.A.	1.001

ANOVA: Single Factor

Source of Variation	SS	df	MS	F	P value	F _{crit}
Between Groups	0.092	5	0.0184	6.12	0.00078	2.60
Within Groups	0.075	25	0.0030			
Total	0.167	30				



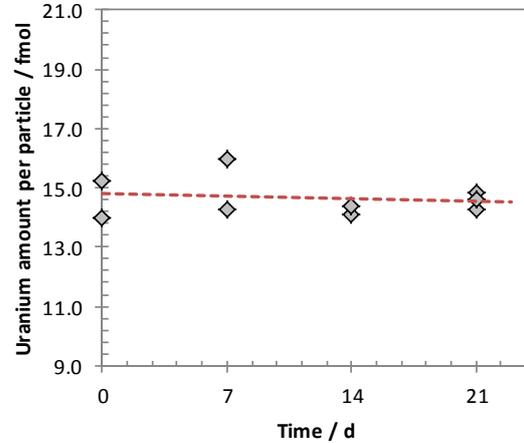
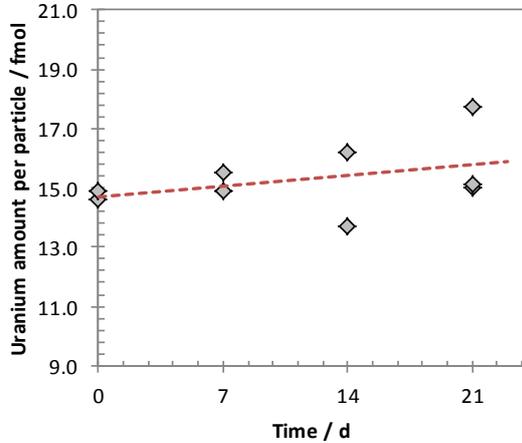
Annex 11. Short-term stability measurement results

4 °C			
t / d	Replicate	Analysis no.	N / fmol
0	1	8	14.591
0	2	12	14.898
7	1	15	14.918
7	2	3	15.489
14	1	17	13.706
14	2	16	16.212
21	1	18	15.007
21	2	10	15.140
21	3	14	17.725

60 °C			
t / d	Replicate	Analysis no.	N / fmol
0	1	9	13.959
0	2	1	15.238
7	1	2	15.996
7	2	6	14.277
14	1	4	14.120
14	2	7	14.400
21	1	13	14.269
21	2	5	14.856
21	3	11	14.616

Linear regression			
b_1	0.052	14.69	b_0
$s(b_1)$	0.046	0.65	$s(b_0)$
r^2	0.157	1.11	$s(y)$
F	1.301	7	df
SS	1.600	8.61	residual SS
t	1.14		
$t_{crit,95\%}$	2.36	no significant difference	
$t_{crit,99\%}$	3.50	no significant difference	

Linear regression			
b_1	-0.012	14.78	b_0
$s(b_1)$	0.028	0.40	$s(b_0)$
r^2	0.026	0.68	$s(y)$
F	0.187	7	df
SS	0.086	3.22	residual SS
t	0.43		
$t_{crit,95\%}$	2.36	no significant difference	
$t_{crit,99\%}$	3.50	no significant difference	



Annex 12. Uranium isotopic composition in IRMM-2329P

IRMM-2329P uranium isotopics		
<p>IRMM-2329P uranium isotopics Author: European Commission Calculations based on values given in the measurement certificate (#3867)</p> <p>Model Equation:</p> <p>{ Isotope amount ratios }</p> $R_{234/238};$ $R_{235/238};$ $R_{236/238};$ $R_{238/238};$ <p>{ Isotope amount fractions }</p> $\Sigma R = R_{234/238} + R_{235/238} + R_{236/238} + R_{238/238};$ $X_{234} = R_{234/238} / \Sigma R;$ $X_{235} = R_{235/238} / \Sigma R;$ $X_{236} = R_{236/238} / \Sigma R;$ $X_{238} = R_{238/238} / \Sigma R;$ <p>{ Molar mass }</p> $M = X_{234} * M_{234} + X_{235} * M_{235} + X_{236} * M_{236} + X_{238} * M_{238};$ <p>{ Isotope mass fractions }</p> $W_{234} = X_{234} * M_{234} / M;$ $W_{235} = X_{235} * M_{235} / M;$ $W_{236} = X_{236} * M_{236} / M;$ $W_{238} = X_{238} * M_{238} / M;$		
List of Quantities:		
Quantity	Unit	Definition
$R_{234/238}$		U-234/U-238 isotope amount ratio of particles
$R_{235/238}$		U-235/U-238 isotope amount ratio of particles
$R_{236/238}$		U-236/U-238 isotope amount ratio of particles
$R_{238/238}$		U-238/U-238 isotope amount ratio of particles
ΣR		Sum of isotope amount ratios
X_{234}		U-234 amount fraction of particles
X_{235}		U-235 amount fraction of particles
X_{236}		U-236 amount fraction of particles
X_{238}		U-238 amount fraction of particles
M	g/mol	Molar mass of sample
M_{234}	g/mol	Molar mass of U-234
M_{235}	g/mol	Molar mass of U-235
M_{236}	g/mol	Molar mass of U-236
M_{238}	g/mol	Molar mass of U-238
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IRMM-2329P uranium isotopics		
Quantity	Unit	Definition
W_{234}		U-234 mass fraction of particles
W_{235}		U-235 mass fraction of particles
W_{236}		U-236 mass fraction of particles
W_{238}		U-238 mass fraction of particles
$R_{234/238}$:	Type B normal distribution Value: 0.000340825 Expanded Uncertainty: 0.000000186 Coverage Factor: 2	
U-234/U-238 isotope amount ratio from certificate of reference measurement (#3867)		
$R_{235/238}$:	Type B normal distribution Value: 0.033902 Expanded Uncertainty: 0.000012 Coverage Factor: 2	
U-235/U-238 isotope amount ratio from certificate of reference measurement (#3867) See certificate #3867 (S Richter)		
$R_{236/238}$:	Type B normal distribution Value: 0.000030207 Expanded Uncertainty: 0.000000124 Coverage Factor: 2	
U-236/U-238 isotope amount ratio from certificate of reference measurement (#3867)		
$R_{238/238}$:	Constant Value: 1	
U-238/U-238 ratio is constant (=1)		
M_{234} :	Type B normal distribution Value: 234.0409504 g/mol Expanded Uncertainty: 0.0000012 g/mol Coverage Factor: 1	
Value and uncertainty from AME2016 Chinese Physics C 41 (2017) 030003 https://www-nds.iaea.org/amdc/		
M_{235} :	Type B normal distribution Value: 235.0439282 g/mol Expanded Uncertainty: 0.0000012 g/mol Coverage Factor: 1	
Value and uncertainty from AME2016 Chinese Physics C 41 (2017) 030003 https://www-nds.iaea.org/amdc/		
M_{236} :	Type B normal distribution Value: 236.0455662 g/mol Expanded Uncertainty: 0.0000012 g/mol Coverage Factor: 1	
Value and uncertainty from AME2016 Chinese Physics C 41 (2017) 030003 https://www-nds.iaea.org/amdc/		
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IRMM-2329P uranium isotopics				
M₂₃₈	Type B normal distribution Value: 238.0507870 g/mol Expanded Uncertainty: 0.0000016 g/mol Coverage Factor: 1 Value and uncertainty from AME2016 Chinese Physics C 41 (2017) 030003 https://www-nds.iaea.org/amdc/			
Interim Results:				
Quantity	Value			
ΣR	1.03427303			
Results:				
Quantity	Value	Expanded Uncertainty	Coverage factor	Coverage
X ₂₃₄	329.53·10 ⁻⁶	180·10 ⁻⁹	2.00	95% (t-table 95.45%)
X ₂₃₅	0.032779	11·10 ⁻⁶	2.00	95% (t-table 95.45%)
X ₂₃₆	29.21·10 ⁻⁶	120·10 ⁻⁹	2.00	95% (t-table 95.45%)
X ₂₃₈	0.966863	11·10 ⁻⁶	2.00	95% (t-table 95.45%)
M	237.950847 g/mol	34·10 ⁻⁶ g/mol	2.00	95% (t-table 95.45%)
W ₂₃₄	324.12·10 ⁻⁶	180·10 ⁻⁹	2.00	95% (t-table 95.45%)
W ₂₃₅	0.032378	11·10 ⁻⁶	2.00	95% (t-table 95.45%)
W ₂₃₆	28.97·10 ⁻⁶	120·10 ⁻⁹	2.00	95% (t-table 95.45%)
W ₂₃₈	0.967269	11·10 ⁻⁶	2.00	95% (t-table 95.45%)
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