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JRC REFERENCE MATERIALS REPORT

IRMM-2030: Certification of a Uranium Nitrate Solution Reference Material with a ^{235}U "Enrichment" (*i.e.* $n(^{235}\text{U})/n(\text{U})$) of 6 %

Certified for Isotope Ratios

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Contents

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 (JRC) 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-2030, a uranium nitrate solution isotopic reference material with an isotope amount fraction $n(^{235}\mathrm{U})/n(\mathrm{U})$ of 6 %, commonly *called a ^{235}U enrichment of 6 %*. The project was a collaboration of JRC-G.2 with Urenco/Germany and the Analytical Services Laboratory of the JRC-G.II.8 Unit in Karlsruhe/Germany.

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The authors very much appreciate the suggestion by Reimund Lammers from Urenco/Gronau in Germany to prepare a uranium nitrate solution isotopic reference material with an isotope amount fraction $n(^{235}U)/n(U)$ of 6 %, commonly called *a ^{235}U enrichment of 6 %* in response to a general need in nuclear industry.

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Abstract

This report describes the certification of the IRMM-2030 uranium nitrate solution reference material, certified for the uranium isotopic composition. The certified values and their uncertainties were assigned following ISO 17034 [1], ISO Guide 35 [2] and the Guide to the Expression of Uncertainty in Measurement [3].

This certification project was a collaboration between the Joint Research Centre's Unit G.2 in Geel/Belgium, the Urenco facility in Gronau/Germany and the Analytical Services Laboratory of Unit JRC-G.II.8 of the Joint Research Centre in Karlsruhe/Germany.

The IRMM-2030 reference material has been prepared in order to provide a uranium nitrate solution isotopic reference material with an isotope amount fraction $n(^{235}U)/n(U)$ of 6 %, commonly called *a ^{235}U enrichment of 6 %,* in response to a need expressed by nuclear industry. This reference material is particularly needed at enrichment facilities such as Urenco/Germany that typically enrich ^{235}U between 3 %and 5 % but are aiming to enrich uranium up to 6 %. The material was also requested by the International Atomic Energy Agency (IAEA) as a reference material for quality control (QC) purposes.

For the preparation of IRMM-2030, two already characterized uranium base materials were mixed to achieve a ^{235}U enrichment within limits specified by the requestor. The first material was a previously characterized UF_6 material named UREU502648 with a ^{235}U enrichment of about 4.9 %. The UF_6 material was hydrolysed and converted to a uranyl nitrate solution. The second base material is a solution prepared from the certified reference material NBL U500 with a ^{235}U enrichment of about 50 %. By the use of this highly enriched second base material, the lower ^{234}U, ^{235}U and ^{236}U abundances of the first base material were increased to achieve the specified ^{234}U, ^{235}U and ^{236}U abundances for IRMM-2030 simultaneously in one mixing step.

The isotopic composition of the base solution IRMM-2030 was measured and certified using the TIMS/MTE method at JRC-G.2 in Geel. After dilution and dispensing into screw-cap quartz ampoules, the isotope ratios were verified for three randomly stratified chosen ampoules internally at JRC-G2 in Geel. The Analytical Service Laboratory at JRC-G.II.8 in Karlsruhe verified independently another set of three randomly stratified chosen ampoules.

The isotopic reference material IRMM-2030 with an enrichment of 6 % for ^{235}U is part of a systematic program of JRC-G.2 to supply isotope reference materials to the safeguards community and to partners and stakeholders. IRMM-2030 can also be considered as an extension of the IRMM-2019-2029 uranium solution reference materials [4, 5] with ^{235}U enrichments from 0.16 % - 5.0 % towards a ^{235}U enrichment of 6 %. These reference materials are intended for the calibration of instruments and methods, quality control purposes. As with any certified reference material, they can also be used for validation studies.

The following values were assigned to IRMM-2030:

URANIUM NITRATE SOLUTION IRMM-2030		
	Isotope amount ratio	
	Certified value 1) [mol/mol]	Uncertainty 2) [mol/mol]
$n(^{234}U)/n(^{238}U)$	0.00052968	0.00000023
$n(^{235}U)/n(^{238}U)$	0.062659	0.000020
$n(^{236}U)/n(^{238}U)$	0.000066513	0.000000027
	Isotope amount fraction	
	Certified value 3) [mol/mol]	Uncertainty 2) [mol/mol]
$n(^{234}U)/n(U)$	0.00049817	0.00000022
$n(^{235}U)/n(U)$	0.058931	0.000018
$n(^{236}U)/n(U)$	0.000062556	0.000000025
$n(^{238}U)/n(U)$	0.940508	0.000018
	Isotope mass fraction	
	Certified value 3) 4) [g/g]	Uncertainty 2) [g/g]
$m(^{234}U)/m(U)$	0.00049015	0.00000021
$m(^{235}U)/m(U)$	0.058231	0.000018
$m(^{236}U)/m(U)$	0.000062076	0.000000025
$m(^{238}U)/m(U)$	0.941217	0.000017
	Molar mass	
	Certified value 3) 4) [g/mol]	Uncertainty 2) [g/mol]
$M(U)$	237.871466	0.000053

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 *M. Wang, W.J. Huang, F.G. Kondev, G.Audi, S. Naimi, "The AME 2020 atomic mass evaluation", Chinese Physics C 45(3): 030003-1 – 030003-512 (2020):*

$M(^{234}U)$ = (234.0409503 ± 0.0000024) g / mol (k = 2)

$M(^{235}U)$ = (235.0439281 ± 0.0000024) g / mol (k = 2)

$M(^{236}U)$ = (236.0455661 ± 0.0000024) g / mol (k = 2)

$M(^{238}U)$ = (238.0507869 ± 0.0000032) g / mol (k = 2)

1 Introduction

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 (JRC), Geel in Belgium (formerly known as the "Institute for Reference Materials and Measurements" (IRMM)), provides a wide range of nuclear certified reference materials (CRMs) to the safeguards authorities and the nuclear industry. This is an obligation under the Euratom treaty, where the need for isotope standards is explicitly mentioned, acknowledging their importance for the measurements of nuclear materials. For accurate mass spectrometric measurements in nuclear material accountancy and nuclear safeguards, suitable CRMs are needed to validate measurement procedures and to calibrate instruments.

The suggestion to prepare IRMM-2030 originates from stakeholder's communications and feedback. Particularly, the enrichment company Urenco/Germany expressed the need for IRMM-2030 in support to satisfy their legal obligation towards license application for a uranium "enrichment" (*i.e.* ($n(^{235}U)/n(U)$) of 6 %, and which will be in the remainder of the document referred to as *a ^{235}U enrichment of 6 %* . A licence for an authorized ^{235}U enrichment of 6 % is considered to be beneficial for enrichment facilities for two main reasons. Firstly, it is important to demonstrate compliance with regulations, and secondly it seems to be often more economical to use a 6% enriched material as a blending partner to produce a customer specified material with a 4 % - 5 % enrichment. According to the IAEA, several enrichment facilities have a license for a ^{235}U enrichment up to 6 %. As a consequence, the IAEA receives safeguards samples for analysis with more than 5 % enrichment for ^{235}U coming from these facilities, and for that reason the IAEA also expressed a strong interest to JRC-Geel to receive such a reference material for quality control purposes.

Therefore, following the needs from the nuclear industry and the IAEA, the IRMM-2030 has been prepared according to specifications given by Urenco/Germany and confirmed by the IAEA. The specifications were given by ranges for the isotope mass fractions of ^{234}U, ^{235}U and ^{236}U as shown in Table 1.

Table 1. Specification for mass fractions of ^{234}U, ^{235}U and ^{236}U for IRMM-2030.

	$m(^{234}U)/m(U)$, %	$m(^{235}U)/m(U)$, %	$m(^{236}U)/m(U)$, %
Lower Limit	0.0440	5.700	0.0020
Average	0.0518	5.825	0.0060
Upper Limit	0.0595	5.950	0.0100

Calculations at JRC-Geel showed that these specifications could be realized by a two component mixture, consisting of an available uranium material with a mass fraction of about 4.9 % ^{235}U and rather low isotope mass fractions for ^{234}U and ^{236}U, and the well-known reference material U500 from NBL. The NBL 500 with a mass fraction of about 50% ^{235}U appeared to be the best choice as the second base material in order to increase the mass fractions of ^{234}U, ^{235}U and ^{236}U simultaneously into the specified ranges.

The characterization of the isotopic composition was achieved by mass spectrometric measurements using the standardized TIMS/MTE method [6, 7, 8], and using the gravimetrically prepared reference materials IRMM-074/10 [9] and IRMM-075/1 [10] as a calibrant (*i.e.* reference material for calibration purposes) and for quality control (QC) purposes, respectively. The certificates of the IRMM-074 and IRMM-075 series are given in Annex 1.

In addition to the sample with a total uranium mass of 1 g ordered by Urenco/Germany, a set of 90 screw-cap quartz ampoules was filled with a diluted solution with a mass fraction of about 2 mg/g, which will be available for sale. IRMM-2030 constitutes an extension of the IRMM-2019-2029 uranium solution reference materials [4, 5] towards a ^{235}U enrichment of 6 %.

2 The Preparation of IRMM-2030

The new reference material IRMM-2030 was prepared by mixing two base materials. The first was a UF_6 material named UREU502648 (also internally known as BC02967) with a ^{235}U mass fraction of about 4.9 % and rather low mass fractions for the minor isotopes ^{234}U and ^{236}U. The isotopic mass fractions are shown in Table 2 and Annex 2. The second base material is the reference material U500 from NBL with a ^{235}U mass fraction of about 50 %, the mass fractions are also given in Table 2 and Annex 2.

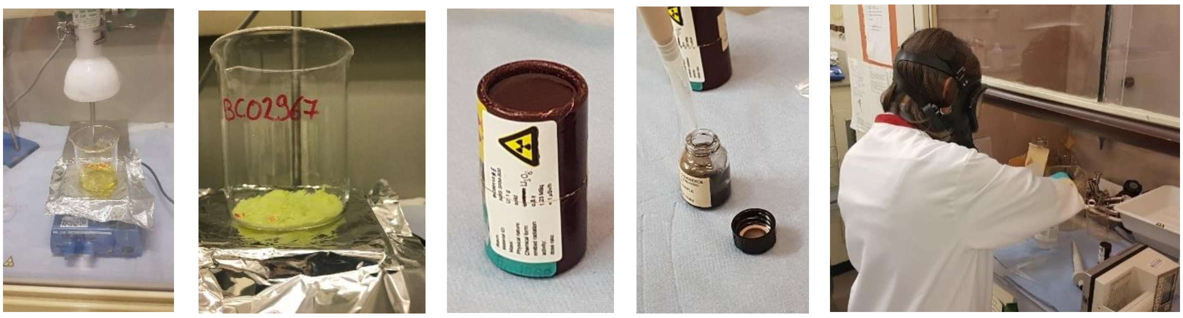
Table 2. Isotopic mass fractions for the base materials UREU502648 and NBL CRM U500 used for IRMM-2030. Uncertainties are given in brackets with a coverage factor of $k = 2$ and apply to the last two digits of the measurement result.

	$m(^{234}U)/m(U)$	$m(^{235}U)/m(U)$	$m(^{236}U)/m(U)$	$m(^{238}U)/m(U)$
UREU502648	0.00038569(14)	0.048414(12)	0.000046497(23)	0.951154(12)
NBL U500	0.005126(8)	0.49383(50)	0.000754(3)	0.50029(50)

The first component for the mixture, the UF_6 material named UREU502648, was hydrolysed and converted into a uranyl nitrate solution. The solution was dried under a heat lamp as much as possible without taking the risk that dried parts of the nitrate could start flaking and being released from the bottom of the beaker. Neither the exact stoichiometry nor the exact uranium mass of the uranyl nitrate is known. The dried nitrate was weighed and dissolved in nitric acid towards an estimated lower limit for the mass fraction of 90 mg/g.

The second component was 1 g of uranium from NBL U500 in the form of U_3O_8. The oxide was dissolved volumetrically by adding about 8 mL of concentrated HNO_3. The mass fraction of U for the NBL CRM U500 solution was also estimated to be about 90 mg/g, but not accurately known. Images of the two components used for the preparation of IRMM-2030 are shown in Figure 1 below.

Figure 1. The two components used for the preparation of IRMM-2030. From the left to right: drying of UREU502648 (also internally known as BC02967) under a heat lamp, dried uranyl nitrate of UREU502648, NBL U500 packaging, opened NBL U500 vial, dissolving NBL U500 inside a fume hood.



The solutions for the two components UREU502648 and NBL U500 with about similar mass fractions were mixed with a ratio of about 44 for the mass of UREU502648 versus the mass of NBL U500. The mixture is labelled “Urenco S7” and has a uranium mass fraction of about 90 mg/g as well. The mixing process was performed carefully in seven consecutive steps to avoid exceeding the specified target values for the isotope mass fractions. A small portion of NBL U500 solution was added into the UREU502648 solution at each step followed by a subsequent determination of the isotopic composition by TIMS/MTE, as it was not possible to derive the isotopic composition of the mixture using the known mixing proportions. Therefore it was decided to perform a direct characterization of the isotopic composition for the mixture by TIMS/MTE at each mixing step. In addition, the determination of the total uranium mass fraction for the mixture, as requested by the customer and needed for the further processing of IRMM-2030, was performed by IDMS/TIMS using the ^{235}U spike IRMM-054 (certificate presented in Annex 1).

3 Characterization

The final mixture "Urenco S7" of the two components UREU502648 and NBL CRM U500 with a mass fraction of about 90 mg/g was divided into seven subsamples and dispensed into quartz screw-cap ampoules, with two of the subsamples designated for the customer (Urenco/Germany), one ampoule for TIMS/MTE measurements of the isotopic composition, one ampoule for IDMS/TIMS measurements, one ampoule for further dilution and dispensing of IRMM-2030, and two ampoules for archiving.

The characterization of the major ratios $n(^{235}\mathrm{U})/n(^{238}\mathrm{U})$ and the minor ratios $n(^{234}\mathrm{U})/n(^{238}\mathrm{U})$ and $n(^{236}\mathrm{U})/n(^{238}\mathrm{U})$ was performed at JRC-Geel using the TIMS/MTE method according to [6, 7, 8]. The gravimetrically prepared reference materials IRMM-074/10 [9] and IRMM-075/1 [10] were used as a calibrant for the mass fractionation correction and for quality control (QC) purposes, respectively. The MTE method has been recently upgraded at JRC-Geel for the use of Faraday cup amplifiers with 10^{13} Ω resistors in order to reduce the signal-to-noise ratio for measurement of the minor isotopes $^{234}\mathrm{U}$ and $^{236}\mathrm{U}$, leading to lower uncertainties for the minor ratios $n(^{234}\mathrm{U})/n(^{238}\mathrm{U})$ and $n(^{236}\mathrm{U})/n(^{238}\mathrm{U})$ [11]. The upgrade of the method requires longer integration and waiting times for the measurement of the minor isotopes and an improved control of the instrument mass calibration. The details of the upgraded MTE method and QC measurement results using the IRMM-075 series are described in [11] and in the draft revised version of the ASTM C1832 standard document, which is expected to be released in 2021.

The determination of the mass fraction of the sample "Urenco-S7" was achieved by IDMS/TIMS/MTE using the $^{235}\mathrm{U}$ spike reference material IRMM-054. The result is not certified, but a report of the analysis has been issued to Urenco/Germany. Since a relative uncertainty of about 0.5 % was considered sufficient by the customer and also for the further processing at JRC-Geel, *i.e.* dilution and dispensing of IRMM-2030 into screw cap vials, only one blend of a fraction of the "Urenco-S7" sample with the spike IRMM-054 was deemed necessary. The result of the IDMS was a value for the mass fraction of 97.93(30) mg/g (k = 2) for the "Urenco-S7" sample, the calculation is shown in Annex 3.

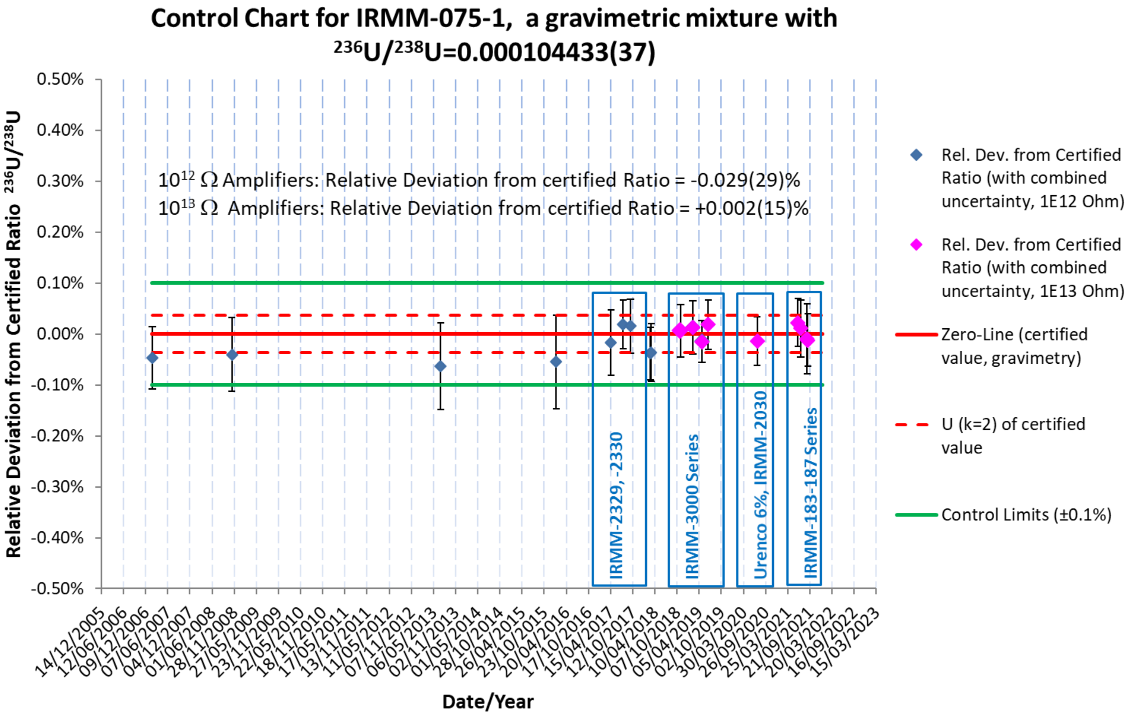
As a consequence, a factor of about 49 was calculated for the dilution of one subsample of "Urenco S7" towards a mass fraction of 2 mg/g and subsequent dispensing into 95 quartz screw-cap vials to be used as reference material IRMM-2030. For process control measurements of the isotopic composition, three ampoules were randomly selected. The results for the isotopic composition measurements of the original mixture "Urenco-S7" and the three selected vials are shown in Table 3. The results for the three vials are in agreement with the results for the isotopic composition of "Urenco S7", but were not included for the calculation of the certified isotopic composition following the initial planning. The isotopic composition of "Urenco S7" is subsequently used for the certification of IRMM-2030.

Table 3. Isotope ratios for mixture "Urenco S7"of UREU502648 versus NBL CRM U500, determined using the TIMS/MTE method according to ASTM C1832. The number of replicate TIMS/MTE measurements (n) is given. Uncertainties are given with a coverage factor of k = 2 and apply to the last two digits of the measurement result.

Sample	$n(^{234}\mathrm{U})/n(^{238}\mathrm{U})$	$n(^{235}\mathrm{U})/n(^{238}\mathrm{U})$	$n(^{236}\mathrm{U})/n(^{238}\mathrm{U})$
Urenco S7 (n = 13)	0.00052968(23)	0.062659(20)	0.000066513(27)
Ampoule 7 (n = 3)	0.00052966(27)	0.062643(24)	0.000066526(43)
Ampoule 43 (n = 3)	0.00052949(37)	0.062643(30)	0.000066486(30)
Ampoule 78 (n = 3)	0.00052966(46)	0.062666(35)	0.000066580(84)

Measurements of the gravimetrically prepared reference material IRMM-075/1 were performed for QC purposes at JRC-Geel in particular for the measurement of $n(^{236}\mathrm{U})/n(^{238}\mathrm{U})$ ratios using the new 10^{13} Ω amplifiers for measuring $^{236}\mathrm{U}$. The results are shown in Figure 2. The data go beyond those already been published in [11], and confirm the accuracy of the $n(^{236}\mathrm{U})/n(^{238}\mathrm{U})$ ratios for the certification of "Urenco S7" and IRMM-2030.

Figure 2. QC measurements for the $n(^{236}U)/n(^{238}U)$ ratios for IRMM-075/1 using the new 10^{13} Ω amplifiers.



In order to comply with ISO 17034 [1], external verification measurements of the isotopic composition were performed by an experienced laboratory (Analytical Services Laboratory of JRC-GII.8 in Karlsruhe/Germany). The results are shown in Table 4 and are in agreement with the isotopic composition shown for “Urenco S7” in Table 3.

Table 4. Isotope ratios for IRMM-2030 measured by the Analytical Services Laboratory of JRC-GII.8 in Karlsruhe/Germany. The number of (successfully) measured replicates using TIMS is given (n). Uncertainties are given in brackets with a coverage factor of k = 2 and apply to the last two digits of the measurement result.

Sample	$n(^{234}U)/n(^{238}U)$	$n(^{235}U)/n(^{238}U)$	$n(^{236}U)/n(^{238}U)$
Ampoule 8 (n = 4)	0.00052954(43)	0.062643(18)	0.00006648(22)
Ampoule 41 (n = 3)	0.00052943(45)	0.062632(19)	0.00006644(19)
Ampoule 67 (n = 2)	0.00052944(60)	0.062636(22)	0.00006649(37)
Average of all individual (successful) replicate measurements for the ampoules 8, 41, 67 (total number n = 9)	0.00052948(22)	0.062638(19)	0.000066468(61)
Relative Difference (%) of average from “Urenco S7” (Table 3)	-0.037(60)%	-0.034(44)%	-0.07(10)%

4 Homogeneity Assessment

A key requirement for any reference material aliquoted into units is equivalence between those units. ISO 17034 [1] requires reference material (RM) producers to quantify the between-unit variation. This aspect is covered in between-unit homogeneity studies.

A homogeneity assessment was considered unnecessary due to the homogeneous nature of the isotopic composition of the uranium nitrate solutions. Evidence for this is the certifications of the series IRMM-019-029 and IRMM-2019-2029, in which the uncertainty contribution from the homogeneity test was demonstrated at the level of the measurement uncertainty and the expected trends with the values of the isotope ratios [4, 5].

5 Stability Assessment

5.1 Short-term stability study

The IRMM-2030 reference material consists of uranium nitrate solution isotopic reference materials in HNO_3 (*c* = 1 mol/L) contained in screw-cap quartz ampoules. Since the isotopic composition is independent of the temperature, there is no impact from transportation on the uranium isotopic composition. Therefore no short term stability study was performed and the materials can be dispatched without further precautions under ambient conditions, respecting fissile and radioactive material transport regulations.

5.2 Long-term stability study

A long term stability assessment is considered to be unnecessary, due to the stable nature of the uranium isotope ratios in uranium nitrate solutions. This has been demonstrated in the course of the certification of the IRMM-019-029 and IRMM-2019-2029 series [4, 5], where no significant trends were observed for isotope ratios over a time period of 10 years. Therefore, the validity of the certificate is given for 10 years.

6 Value assignment

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

Usually the assigned uncertainties consist of uncertainties relating to characterisation, u_{char} (section 3), potential within-unit and between-unit inhomogeneities, combined and expressed as u_{hom} (section 4), potential degradation during transport, u_{trn} (section 5.1), and potential degradation during storage, u_{lts} (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:

$$\boldsymbol{U}_{\mathbf{CRM}} = \boldsymbol{k} \cdot \sqrt{\boldsymbol{u}_{\mathbf{char}}^2 + \boldsymbol{u}_{\mathbf{hom}}^2 + \boldsymbol{u}_{\mathbf{trn}}^2 + \boldsymbol{u}_{\mathbf{lts}}^2} \qquad \text{Equation 1}$$

As explained before in sections 4 and 5, no assessment of the homogeneity nor of the stability were deemed necessary for the highly enriched uranium nitrate solutions. Therefore the uncertainties u_{hom}, u_{trn} and u_{lts} are considered zero. Consequently, only the uncertainties from the characterization u_{char} have to be taken into account for the certification of the uranium isotope amount ratios in IRMM-2030. The uncertainty budgets for the certified isotope ratios are shown in Table 5.

Table 5. Uncertainty budgets for the certified isotope ratios of IRMM-2030. Uncertainties are given in brackets with a coverage factor of k = 2 and apply to the last two digits of the measurement result.

	$n(^{235}\text{U})/n(^{238})\text{U}$	$n(^{234}\text{U})/n(^{238})\text{U}$	$n(^{236}\text{U})/n(^{238})\text{U}$
Certified ratio	0.062659(20	0.00052968(23)	0.000066513(27)
Relative Uncertainty contribution (in %)			
IRMM-074, certified $n(^{235}\text{U})/n(^{238})\text{U}$	26.2	23.7	7.1
IRMM-074, measured $n(^{235}\text{U})/n(^{238})\text{U}$	3.4	3.0	0.9
IRMM-2030, measured $n(^{235}\text{U})/n(^{238})\text{U}$	29.6	26.8	8.0
IRMM-2030, measured $n(^{234}\text{U})/n(^{238})\text{U}$		9.5	
IRMM-2030, measured $n(^{236}\text{U})/n(^{238})\text{U}$			73.0
External uncertainty for MTE [4, 5]	40.9	37.0	11.0

6.1 Uranium isotope amount ratios, isotope amount fractions, isotope mass fractions and molar mass

The results of the characterization of the uranium isotope amount ratios for the IRMM-2030 reference material are presented within section 3 in Table 3 (sample "Urenco S7"). These results are assigned as the certified values. The uranium isotope amount ratios are given in mol·/ mol.

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:

$$\boldsymbol{n}(\,^{x}\mathbf{U})/\boldsymbol{n}(\mathbf{U}) = \frac{\boldsymbol{n}\left(^{x=(234,;235,;236,;238)}\,\mathbf{U}\right)/\boldsymbol{n}\left(^{238}\mathbf{U}\right)}{\sum_{i=\{234;235;236;238\}}^{i} \boldsymbol{n}(\,^{i}\mathbf{U})/\boldsymbol{n}(^{238}\mathbf{U})} \qquad \text{Equation 2}$$

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

$$\boldsymbol{M}(\mathbf{U}) = \sum_{i=\{234;235;236;238\}}^{i} \boldsymbol{n}(\,^{i}\mathbf{U})/\boldsymbol{n}(\mathbf{U}) \cdot \boldsymbol{M}(\,^{i}\mathbf{U}) \qquad \text{Equation 3}$$

For the calculation of the uranium molar mass, the molar mass of the individual isotopes of uranium (^{234}U, ^{235}U, ^{236}U and ^{238}U) have been taken from the most recent atomic mass evaluation (AME 2020, [13]).

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 in the material:

$$\boldsymbol{m}(\,^{x}\mathbf{U})/\boldsymbol{m}(\mathbf{U}) = \frac{\boldsymbol{n}\left(^{x=(234:235:236:238)}\mathbf{U}\right)/\boldsymbol{n}(\mathbf{U})\cdot \boldsymbol{M}(\,^{x}\mathbf{U})}{\boldsymbol{M}(\mathbf{U})} \qquad \text{Equation 4}$$

The certified and calculated derived values related to the uranium isotopic composition for IRMM-2030 are summarised in Table 6. The calculation of the certified isotope amount fractions, isotope mass fractions and the molar mass is shown in Annex 4.

The isotope mass fractions $m(^{234}\mathrm{U})/m(\mathrm{U})$, $m(^{235}\mathrm{U})/m(\mathrm{U})$ and $m(^{236}\mathrm{U})/m(\mathrm{U})$ of IRMM-2030 are well within the requested limits presented in Table 1. Therefore it can be concluded that the applied mixing procedure was successful.

Table 6. Certified Values for IRMM-2030. Uncertainties are given in brackets with a coverage factor of k = 2 and apply to the last two digits of the measurement result.

	$n(^{234}\mathrm{U})/n(^{238}\mathrm{U})$	$n(^{235}\mathrm{U})/n(^{238}\mathrm{U})$	$n(^{236}\mathrm{U})/n(^{238}\mathrm{U})$	
IRMM-2030	0.00052968(23)	0.062659(20)	0.000066513(27)	
	$n(^{234}\mathrm{U})/n(\mathrm{U})$	$n(^{235}\mathrm{U})/n(\mathrm{U})$	$n(^{236}\mathrm{U})/n(\mathrm{U})$	$n(^{238}\mathrm{U})/n(\mathrm{U})$
IRMM-2030	0.00049817(22)	0.058931(18)	0.000062556(25)	0.940508(18)
	$m(^{234}\mathrm{U})/m(\mathrm{U})$	$m(^{235}\mathrm{U})/m(\mathrm{U})$	$m(^{236}\mathrm{U})/m(\mathrm{U})$	$m(^{238}\mathrm{U})/m(\mathrm{U})$
IRMM-2030	0.00049015(21)	0.058231(18)	0.000062076(25)	0.941217(17)
	Molar Mass (g/mol)			
IRMM-2030	237.871466(53)			

7 Metrological traceability and commutability

7.1 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-2030 are traceable to the International SI via the certified $n(^{235}U)/n(^{238}U)$ isotope ratio of the certified reference material IRMM-074.

7.2 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 [12] 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.

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.

8 Instructions for use

8.1 Safety information

The IRMM-2030 reference material contains low-level 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. The α-activity of each unit of IRMM-2030 is about 1300Bq.

8.2 Storage conditions

The material 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.

8.3 Use of the certified values

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

8.4 Use as a calibrant (*i.e.* reference material used for calibration purposes)

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

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 [14]:

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}): $u_\Delta = \sqrt{u_{\mathrm{meas}}^2 + u_{\mathrm{CRM}}^2}$.
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 $\Delta_{meas} \le U_\Delta$ then no significant difference exists between the measurement result and the certified value, at a confidence level of approximately 95 %.

8.5 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] 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

[5] 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", Journal of Radioanalytical and Nuclear Chemistry, 318:1359–1368 (2018)

[6] S. Richter, S. A. Goldberg, “Improved Techniques for High Accuracy Isotope Ratio Measurements of Nuclear Materials using Thermal Ionization Mass Spectrometry”, International Journal of Mass Spectrometry, 229: 181-197 (2003)

[7] 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)

[8] ASTM Standard Document C1832, available through the ASTM website: https://www.astm.org/Standards/C1832.htm

[9] S. Richter, A. Alonso, Y. Aregbe, R. Eykens, F. Kehoe, H.Kühn, N. Kivel, A. Verbruggen, R. Wellum, P.D.P. Taylor, “A New Series of Uranium Isotope Reference Materials for Investigating the Linearity of Secondary Electron Multipliers in Isotope Mass Spectrometry”, International Journal of Mass Spectrometry, 281: 115-125 (2009)

[10] S. Richter, A. Alonso, Y. Aregbe, R. Eykens, U. Jacobsson, F. Kehoe, H. Kühn, A. Verbruggen, R. Wellum, “Certification of a new Series of Gravimetrically Prepared Synthetic Reference Materials for $n(^{236}U)/n(^{238}U)$ Isotope Ratio Measurements”, Proceedings of the 11th International Conference on Accelerator Mass Spectrometry, Rome, Italy, Sept. 2008, Nuclear Instruments and Methods in Physics Research B 268: 956-959 (2010)

[11] S. Richter, “Implementation of Faraday Cup Amplifiers with $10^{13}\Omega$ Resistors for TIMS measurements at JRC-G.2 in Geel”, INMM Annual Meeting 2019, Palm Desert, CA, United States, July 2019.

[12] 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)

[13] M. Wang, W.J. Huang, F.G. Kondev, G.Audi, S. Naimi, “The AME 2020 atomic mass evaluation”, Chinese Physics C 45(3): 030003-1 – 030003-512 (2020)

[14] T. Linsinger, “Comparison of a measurement result with the certified value”, European Reference Materials, Application Note 1 (2010)

List of abbreviations and definitions

ASTM	American Society for Testing and Materials
CRM	Certified reference material
EC	European Commission
IAEA	International Atomic Energy Agency
IDMS	Isotope dilution mass spectrometry
JRC	Joint Research Centre
k	Coverage factor
M	Molar mass
MTE	Modified total evaporation
NBL	New Brunswick Laboratory Program Office
SGAS	Safeguards Analytical Services of the IAEA
SI	International system of units
TIMS	Thermal ionisation mass spectrometry
U	Expanded uncertainty
u	Standard uncertainty
u_{char}	Standard uncertainty due to material characterisation
u_{CRM}	Combined standard uncertainty of the certified value
u_{hom}	Standard uncertainty of the homogeneity study
u_{lts}	Standard uncertainty of the long-term stability
u_{trn}	Standard uncertainty due to short-term stability (transport)
UF_6	Uranium hexafluoride
VIM	International vocabulary on metrology

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Annexes

Annex 1. Certificates of reference materials, IRMM-074, IRMM-075, IRMM-054

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 – 074

CERTIFICATE OF ANALYSIS

Uranium in nitric acid solution

Code number	Certified isotope amount ratios[1)] [mol/mol]		
	$n(^{233}U)/n(^{235}U)$ U = 0.025 % (relative) [2)]	$n(^{233}U)/n(^{238}U)$ U = 0.025 % (relative) [2)]	$n(^{235}U)/n(^{238}U)$ U = 0.015 % (relative) [2)]
IRMM-074/1	1.02685	1.02711	1.000254
IRMM-074/2	0.307993	0.308072	1.000258
IRMM-074/3	0.0102288	0.0102314	1.000259
IRMM-074/4	0.00307358	0.00307437	1.000259
IRMM-074/5	0.00103061	0.00103088	1.000259
IRMM-074/6	0.000307778	0.000307858	1.000259
IRMM-074/7	0.000102603	0.000102629	1.000259
IRMM-074/8	0.0000308011	0.0000308091	1.000259
IRMM-074/9	0.0000081587	0.0000081608	1.000259
IRMM-074/10	0.00000101886	0.00000101913	1.000259

[1)] The certified values are traceable to the International System of units (SI). The reference date for the certified values is June 2005.
[2)] The uncertainty (U) is the relative 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 10 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 2010.

Geel, September 2019

Signed: 18 SEP. 2019

Dr. Arjan Plompen
European Commission
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B-2440 Geel, Belgium

DESCRIPTION OF THE MATERIAL

The IRMM-074 is a uranium isotopic Certified Reference Material (CRM) supplied with isotope amount ratios as certified above. The IRMM-074 consists of a set of ten units, each containing approximately 0.2 mg uranium as uranyl nitrate in 2 mL of nitric acid solution in a sealed quartz glass ampoule. The concentration of nitric acid is 1 mol·L^{-1}.

ANALYTICAL METHODS USED FOR CERTIFICATION

The certified values were established by gravimetric mixing of highly enriched ^{233}U, ^{235}U and ^{238}U starting materials and verified by thermal ionisation mass spectrometry (TIMS).

SAFETY INFORMATION

The IRMM-074 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 intended for the verification and correction of non-linearities of the entire mass spectrometer measurement system.

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.

LEGAL NOTICE

NOTE

A technical report on the preparation and certification of IRMM-074 is available on the internet (https://crm.jrc.ec.europa.eu/). A paper copy can be obtained from JRC - Geel on request.

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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 – 075

CERTIFICATE OF ANALYSIS

Uranium in nitric acid solution		
Code number	Isotope amount ratios $n(^{236}U)/n(^{238}U)$	
	Certified value 1) [mol/mol]	Uncertainty 2) [mol/mol]
IRMM-075/1	$1.04433 \cdot 10^{-4}$	$3.7 \cdot 10^{-8}$
IRMM-075/2	$1.14160 \cdot 10^{-5}$	$4.0 \cdot 10^{-9}$
IRMM-075/3	$1.04093 \cdot 10^{-6}$	$3.6 \cdot 10^{-10}$
IRMM-075/4	$1.13742 \cdot 10^{-7}$	$4.0 \cdot 10^{-11}$
IRMM-074/5	$1.06519 \cdot 10^{-8}$	$7.5 \cdot 10^{-12}$
IRMM-075/6	$1.0885 \cdot 10^{-9}$	$6.3 \cdot 10^{-12}$

1) The certified values are traceable to the International System of units (SI). The reference date for the certified values is May 6, 2006.

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 10 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 2007.

Geel, September 2019

Signed: ______________ 18 SEP. 2019

Dr. Arjan Plompen
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G.2 – Standards for Nuclear Safety, Security and Safeguards Unit
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B-2440 Geel, Belgium

DESCRIPTION OF THE MATERIAL

The IRMM-075 is a uranium isotopic Certified Reference Material (CRM) supplied with isotope amount ratios as certified above. The IRMM-075 consists of a set of six units, each containing approximately 1 mg uranium as uranyl nitrate in 1 mL of nitric acid solution in a sealed quartz glass ampoule. The concentration of nitric acid is 1 $mol \cdot L^{-1}$.

ANALYTICAL METHODS USED FOR CERTIFICATION

The certified values were established by gravimetric mixing of highly enriched ^{236}U and ^{nat}U starting materials and verified by thermal ionisation mass spectrometry (TIMS).

SAFETY INFORMATION

The IRMM-075 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 intended for the verification and correction of non-linearities of the entire mass spectrometer measurement system.

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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NOTE

A technical report on the preparation and certification of IRMM-075 is available on the internet (https://crm.jrc.ec.europa.eu/). A paper copy can be obtained from JRC - Geel on request.

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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 – 054

CERTIFICATE OF ANALYSIS

Uranium in nitric acid solution		
	Isotope amount content	
	Certified value [1)] [µmol/g solution]	Uncertainty [2)] [µmol/g solution]
^{235}U	3.9543	0.0012
	Isotope amount ratios	
	Certified value [1)] [mol/mol]	Uncertainty [2)] [mol/mol]
$n(^{234}U)/n(^{235}U)$	0.010684	0.000020
$n(^{236}U)/n(^{235}U)$	0.004490	0.000010
$n(^{238}U)/n(^{235}U)$	0.058065	0.000034

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 a revision of the original certificate of 1999.

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

Geel, August 2019

Signed: 29-8-2019

Dr. Arjan Plompen
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G.2 – Standards for Nuclear Safety, Security and Safeguards Unit
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The following values were assigned (continued):

	Isotopic mass fractions (·100)	
	Value [3)] [g/g]	Uncertainty [4)] [g/g]
$m(^{234}U)/m(U)$	0.9906	0.0020
$m(^{235}U)/m(U)$	93.1138	0.0070
$m(^{236}U)/m(U)$	0.41984	0.00098
$m(^{238}U)/m(U)$	5.4758	0.0054
	Isotopic amount fractions (·100)	
	Value [3)] [mol/mol]	Uncertainty [4)] [mol/mol]
$n(^{234}U)/n(U)$	0.9955	0.0020
$n(^{235}U)/n(U)$	93.1760	0.0068
$n(^{236}U)/n(U)$	0.41834	0.00098
$n(^{238}U)/n(U)$	5.4102	0.0052
	Amount content	
	Value [3)] [µmol/g solution]	Uncertainty [4)] [µmol/g solution]
U	4.2440	0.0013
	Mass fraction	
	Value [3)] [mg/g solution]	Uncertainty [4)] [mg/g solution]
U	0.99818	0.00030
^{235}U	0.92944	0.00028
	Molar mass	
	Value [3)] [g/mol]	Uncertainty [4)] [g/mol]
U	235.20081	0.00015

3) The derived certified values are calculated from the certified amount content of ^{235}U, 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-054 is a uranium spike Certified Reference Material (CRM) supplied with an isotope amount content of ^{235}U and isotope amount ratios as certified above. A unit of IRMM-054 consists of a flame-sealed glass ampoule containing about 5.9 mg uranium in 5 mL of nitric acid solution. The concentration of nitric acid is about 5 $mol \cdot L^{-1}$.

ANALYTICAL METHODS USED FOR CERTIFICATION

The certified values were established by gravimetric preparation and verified by isotope dilution mass spectrometry (IDMS). The isotope ratio measurements were performed on a Thermal Ionisation Mass Spectrometer and calibrated by means of syntheic uranium isotope mixtures.

SAFETY INFORMATION

The IRMM-054 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

This spike Certified Reference Material (CRM) is used as a calibrant to determine the uranium amount content by isotope dilution mass spectrometry (IDMS).

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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Annex 2. Certificates of base materials, UREU502648, NBL U500

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

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

UREU502648
Certificate of Reference Measurement

Customer	
Company	URENCO Deutschland GmbH
Division	Department of Chemistry
Address	Roentgenstrasse 4 D – 48599 Gronau Germany
Requestor	R. Lammers

Sample Information	
Sample type(s)	Uranium Hexafluoride (UF_6) samples contained in P10 Tubes
Date of receipt of sample(s)	23/02/2017 and 11/04/2017
Customer sample(s) identification	1S-Cylinder: UREU502648 P10: UREU409401 and UREU419904 provided to JRC-Geel
Condition of sample(s)	Uranium Hexafluoride (UF_6)

Measurement Results

Analyte	Result[1]	Unit	Method[2]
$n(^{234}U)/n(^{238}U)$	0.00041244(15)	mol / mol	TIMS/MTE
$n(^{235}U)/n(^{238}U)$	0.051551(13)	mol / mol	TIMS/MTE
$n(^{236}U)/n(^{238}U)$	0.000049300(25)	mol / mol	TIMS/MTE

Derived Quantities

Molar Mass	237.901774(37)

Amount fraction (×100)		Mass fraction (×100)	
$n(^{234}U)/n(U)$	0.039205(14)	$m(^{234}U)/m(U)$	0.038569(14)
$n(^{235}U)/n(U)$	4.9002(12)	$m(^{235}U)/m(U)$	4.8414(12)
$n(^{236}U)/n(U)$	0.0046862(24)	$m(^{236}U)/m(U)$	0.0046497(23)
$n(^{238}U)/n(U)$	95.0559(12)	$m(^{238}U)/m(U)$	95.1154(12)

Notes:		
	1	*Uncertainties are given as expanded (k=2) uncertainties according to the ISO Guide to the Expression of Uncertainty (GUM), corresponding to an approximate 95% confidence interval)*
	2	*Measurement method: "Modified Total Evaporation" (MTE), see related publications:* *1.) Richter, S., H Kühn, Y Aregbe, M Hedberg, J Horta-Domenech, K Mayer, E Zuleger, S Bürger, S Boulyga, A Köpf, J Poths, K Matthew, J. Anal. At. Spectrom., 2011, 26,550.* *2.) ASTM C1832*

Date of analysis (dd/mm/yyyy)	31/07-26/09/2017
Date of internal analysis report (dd/mm/yyyy)	03/10/2017
Certification date normalised to (reference date)	03/10/2017at 12:00 h

Backup Files and Raw Data

All results of mass spectrometric measurements for this request are stored in "G:\JRC.G.2\Nuclear Safeguards\Nuclear\TRITON DATA - SHARED\URENCO 2017", in the following way:

For sub-samples 00VCN12BB533 (UREU409401) and 00VCN22BB802 (UREU419904):
"T170807 Urenco 2017 MTE-Turret-2.xls"
For the averages, labelled UREU502648:
"EXTENDED of T170807 Urenco 2017 MTE-Turret-2.xls"
For isotope amount fractions, isotope mass fractions and the molar mass:
"UREU502648 - Urenco 2017.smu"
The Internal Test Report is stored in:
"G:\JRC.G.2\Nuclear Safeguards\Nuclear\TRITON DATA - SHARED\URENCO 2017" and filed as:
" Internal Test Report 4020- Urenco 2017 - F-D-00376rev04.doc"
The External Test Report is stored in:
"G:\JRC.G.2\Nuclear Safeguards\Nuclear\TRITON DATA - SHARED\URENCO 2017" and filed as:
"External Test Report 4020 - Urenco 2017 - F-D-00375rev03doc"

Annexes	
1.	Annex-1, Transport Papers Urenco-JRC Geel, 2 pages
2.	Annex-2, Email from R. Lammers, 21/09/2017, 2 pages
3.	Copy of (internal) Request for Analysis, 1 page
4.	Copy of Internal Test Report 4020, 4 pages
5.	Copy of External Test Report 4020, 4 pages

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

Signature and date:

10/10/17

New Brunswick Laboratory
U.S. Department of Energy

Certificate of Analysis
CRM U500
Uranium Isotopic Standard
10 mg Uranium as U_3O_8

	^{234}U	^{235}U	^{236}U	^{238}U
Atom Percent:	**0.5181**	**49.696**	**0.0755**	**49.711**
Uncertainty:	**±0.0008**	**±0.050**	**±0.0003**	**±0.050**
Weight Percent:	**0.5126**	**49.383**	**0.0754**	**50.029**

This Certified Reference Material (CRM) is primarily intended for the calibration of mass spectrometers used to perform uranium isotopic measurements. The specific purpose of this isotopic standard is for the determination of mass discrimination effects for uranium isotopes being measured under similar analytical conditions. Each unit of CRM U500 consists of approximately 10 milligrams of uranium, in the form of highly purified U_3O_8, contained in a glass bottle.

The indicated uncertainties for the isotopic composition of the CRM are 95% confidence intervals for a single determination. This term can be defined as an approximate two-sigma limit, where sigma is the standard deviation of the measurements data obtained from the material. The uncertainties include allowances for inhomogeneity of the material as well as analytical error.

This CRM was originally issued in 1970 by the National Bureau of Standards (NBS) as Standard Reference Material (SRM) U-500. The measurements made at NBS leading to the certification were performed by E. L. Garner, L. A. Machlan, M.S. Richmond and W.R. Shields. In 1987, the technical and administrative transfer of NBS Special Nuclear SRMs into the NBL CRM Program was coordinated by the NBS Office of Standard Reference Materials and N. M. Trahey, NBL.

The certified isotopic abundance values were determined using a solid-sample thermal ionization mass spectrometer equipped with a Faraday cup detection system. The measured $^{235}U/^{238}U$ values were corrected for mass discrimination effects by intercomparison with synthetic calibration mixtures of similar ^{235}U levels, prepared from high-purity ^{235}U and ^{238}U separated isotopes. The $^{235}U/^{238}U$ value for this standard, 0.9997, is known to at least 0.1%.

The ^{234}U and ^{236}U abundances were determined at NBS by isotope dilution mass spectrometry using high-purity ^{233}U as the spike.

NOTE: NBS Special Publication 260-27 presents further details of the measurements made at NBS which provided the basis for the certification, and is available from the NBS Office of Standard Reference Materials

March 30, 2008
Argonne, Illinois

Jon Neuhoff, Director
New Brunswick Laboratory

www.nbl.doe.gov
Page 1 of 1

(Editorial revision of Certificate dated October 1, 1987)

Annex 3. IDMS of mixture "Urenco S7".

U IDMS IUrenco S7 with IRMM-054 spike

Author: Jakopic

Author:S Richter

simplified equation spike ^{235}U, sample ^{238}U, ratio $^{235}U/^{238}U$ for IDMS equation

certificate IRMM-054: 3.9543 E-06 (12) mol/g solution

1 blend (weighing certificate): E3943

date of IDMS analysis: 23/07/2020, T200723 MTE - Urenco S7 IDMS IA

IA data: Internal Test Report 4706- Urenco 2017 - F-D-00376rev04

the atomic masses according Wang et al. (The AME 2016 atomic mass evaluation (II). Tables, Graphs and References, Chinese Physics C, Vol. 41, No. 3, 2017).

Model Equation:

{---isotope IDMS equation---}

$f_{c235U}(m_x,m_y,R_b)=c_{054}*m_y/m_x*(R_y-R_b)/(R_b-R_{235/238Ux})*(1/R_y);$

$c_{238U}=f_{c235U}(m_x,m_y,R_b)*F_{ExternUncert};$

{---amount content and mass fraction calculations---------------------------------------}

$c_U=c_{238U}/f_{238Ux};$

$\gamma_U=c_U*M_U;$

{-------------------------------mass fractionation correction, K_{corr}-factor 235U/238U---}

$R_y=1/R_{054};$

{---molar mass of uranium in the sample------------------}

$M_U = M_{234U}\cdot f_{234Ux} + M_{235U}\cdot f_{235Ux} + M_{236U}\cdot f_{236Ux} + M_{238U}\cdot f_{238Ux};$

{---amount fractions in the sample----------------------}

$f_{234Ux} = R_{234/238Ux}/\Sigma R_U;$

$f_{235Ux} = R_{235/238Ux}/\Sigma R_U;$

$f_{236Ux} = R_{236/238Ux}/\Sigma R_U;$

$f_{238Ux} = 1/\Sigma R_U;$

$\Sigma R_U = R_{234/238Ux} + R_{235/238Ux} + R_{236/238Ux} + 1;$

List of Quantities:

Quantity	Unit	Definition
c_{238U}	mol/g	measured mean amount content of ^{238}U, sample
c_U	mol/g	amount content of U in sample
$R_{234/238Ux}$	mol/mol	amount ratio $R_{234}/_{238U}$ in sample
$R_{235/238Ux}$	mol/mol	amount ratio $R_{235}/_{238U}$ in sample
$R_{236/238Ux}$	mol/mol	amount ratio $R_{236}/_{238U}$ in sample
f_{234Ux}	mol/mol	amount fraction of ^{234}U sample
f_{235Ux}	mol/mol	amount fraction of ^{235}U in sample
f_{236Ux}	mol/mol	amount fraction of ^{236}U in sample
f_{238Ux}	mol/mol	amount fraction of ^{238}U in sample
M_U	g/mol	molar mass of uranium in sample
M_{234U}	g/mol	atomic mass for ^{234}U
M_{235U}	g/mol	atomic mass for ^{235}U
M_{236U}	g/mol	atomic mass for ^{236}U
M_{238U}	g/mol	atomic mass for ^{238}U
c_{054}	mol/g	amount content of ^{235}U in spike IRMM-054
ΣR_U	mol/mol	sum of ratios for U in sample
R_y	mol/mol	reversed ^{235}U/^{238}U amount ratio in the spike IRMM-054
R_{054}	mol/mol	certified ^{238}U/^{235}U amount ratio in the spike IRMM-054
m_x	g	mass of sample
m_y	g	mass of spike
R_b	mol/mol	amount ratio $R_{235}/_{238U}$ in spike IRMM-054
γ_U	g/g	mass content of U in sample
$F_{ExternUncert}$	mol/mol	External Uncertainty

$R_{234/238Ux}$:
Type B normal distribution
Value: 0.00052968 mol/mol
Expanded Uncertainty: 0.00000023 mol/mol
Coverage Factor: 2

$R_{235/238Ux}$:
Type B normal distribution
Value: 0.062659 mol/mol
Expanded Uncertainty: 0.000020 mol/mol
Coverage Factor: 2

$R_{236/238Ux}$:
Type B normal distribution
Value: 0.000066513 mol/mol
Expanded Uncertainty: 0.000000027 mol/mol
Coverage Factor: 2

M_{234U}: Type B normal distribution
Value: 234.0409504 g/mol
Expanded Uncertainty: 0.0000024 g/mol
Coverage Factor: 2

the atomic masses according Wang et al. (The AME 2016 atomic mass evaluation (II). Tables, Graphs and References, Chinese Physics C, Vol. 41, No. 3, 2017).

M_{235U}: Type B normal distribution
Value: 235.0439282 g/mol
Expanded Uncertainty: 0.0000024 g/mol
Coverage Factor: 2

the atomic masses according Wang et al. (The AME 2016 atomic mass evaluation (II). Tables, Graphs and References, Chinese Physics C, Vol. 41, No. 3, 2017).

M_{236U}: Type B normal distribution
Value: 236.0455662 g/mol
Expanded Uncertainty: 0.0000024 g/mol
Coverage Factor: 2

Wang et al., The AME 2016 atomic mass evaluation, Chinese Physics C Vol. 41, No. 3 (2017) 030003

M_{238U}: Type B normal distribution
Value: 238.0507870 g/mol
Expanded Uncertainty: 0.0000032 g/mol
Coverage Factor: 2

the atomic masses according Wang et al. (The AME 2016 atomic mass evaluation (II). Tables, Graphs and References, Chinese Physics C, Vol. 41, No. 3, 2017).

c_{054}: Type B normal distribution
Value: $3.9543 \cdot 10^{-6}$ mol/g
Expanded Uncertainty: $0.0012 \cdot 10^{-6}$ mol/g
Coverage Factor: 2

IRMM-054 certificate

R_{054}: Type B normal distribution
Value: 0.058065 mol/mol
Expanded Uncertainty: 0.000034 mol/mol
Coverage Factor: 2

IRMM-054certificate

m_x: Type B normal distribution
Value: 0.47600 g
Expanded Uncertainty: 0.00010 g
Coverage Factor: 2

m_y: Type B normal distribution
Value: 5.46178 g
Expanded Uncertainty: 0.00002 g
Coverage Factor: 2

R_b: Type B normal distribution
Value: 0.178626 mol/mol
Expanded Uncertainty: 0.000048 mol/mol
Coverage Factor: 2

$F_{ExternUncert}$: Type B normal distribution
Value: 1 mol/mol
Expanded Uncertainty: .3 %
Coverage Factor: 2

Additional Uncertainty to take care of the fact that only 1 blend was measured instaed of several

Interim Results:

Quantity	Value	Standard Uncertainty
ΣR_U	1.0632552 mol/mol	$10.0 \cdot 10^{-6}$ mol/mol

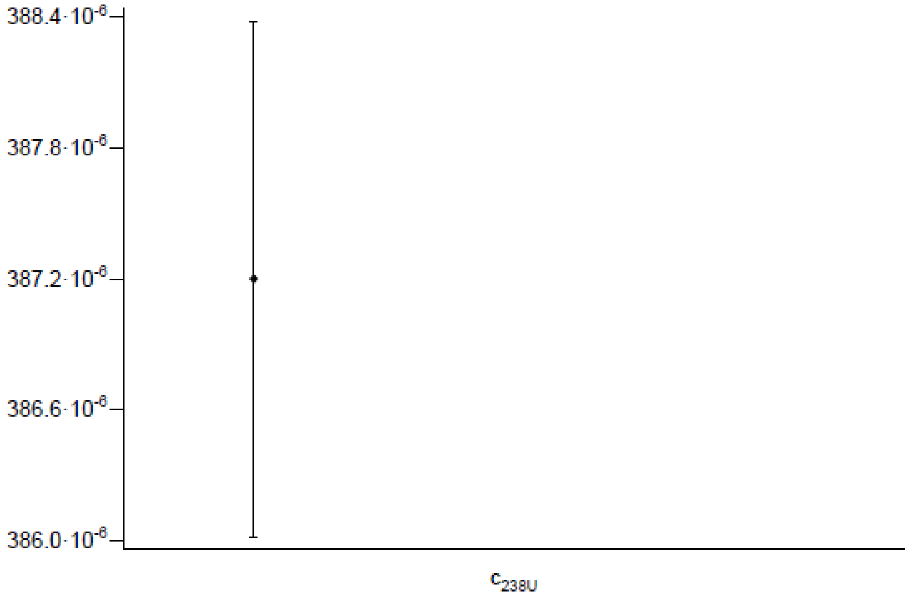
Uncertainty Budgets:

γ_U: **mass content of U in sample**

Quantity	Value	Standard Uncertainty	Distribution	Sensitivity Coefficient	Uncertainty Contribution	Index
$R_{234/238Ux}$	$529.680 \cdot 10^{-6}$ mol/mol	$115 \cdot 10^{-9}$ mol/mol	normal	0.091	$10 \cdot 10^{-9}$ g/g	0.0 %
$R_{235/238Ux}$	0.0626590 mol/mol	$10.0 \cdot 10^{-6}$ mol/mol	normal	0.94	$9.4 \cdot 10^{-6}$ g/g	0.4 %
$R_{236/238Ux}$	$66.5130 \cdot 10^{-6}$ mol/mol	$13.5 \cdot 10^{-9}$ mol/mol	normal	0.091	$1.2 \cdot 10^{-9}$ g/g	0.0 %
M_{234U}	234.04095040 g/mol	$1.20 \cdot 10^{-6}$ g/mol	normal	$210 \cdot 10^{-9}$	$250 \cdot 10^{-15}$ g/g	0.0 %
M_{235U}	235.04392820 g/mol	$1.20 \cdot 10^{-6}$ g/mol	normal	$24 \cdot 10^{-6}$	$29 \cdot 10^{-12}$ g/g	0.0 %
M_{236U}	236.04556620 g/mol	$1.20 \cdot 10^{-6}$ g/mol	normal	$26 \cdot 10^{-9}$	$31 \cdot 10^{-15}$ g/g	0.0 %
M_{238U}	238.05078700 g/mol	$1.60 \cdot 10^{-6}$ g/mol	normal	$390 \cdot 10^{-6}$	$620 \cdot 10^{-12}$ g/g	0.0 %
c_{054}	$3.954300 \cdot 10^{-6}$ mol/g	$600 \cdot 10^{-12}$ mol/g	normal	25000	$15 \cdot 10^{-6}$ g/g	1.0 %
R_{054}	0.0580650 mol/mol	$17.0 \cdot 10^{-6}$ mol/mol	normal	-0.018	$-300 \cdot 10^{-9}$ g/g	0.0 %
m_x	0.4760000 g	$50.0 \cdot 10^{-6}$ g	normal	-0.21	$-10 \cdot 10^{-6}$ g/g	0.5 %
m_y	5.4617800 g	$10.0 \cdot 10^{-6}$ g	normal	0.018	$180 \cdot 10^{-9}$ g/g	0.0 %
R_b	0.1786260 mol/mol	$24.0 \cdot 10^{-6}$ mol/mol	normal	-0.85	$-20 \cdot 10^{-6}$ g/g	1.9 %
$F_{ExternUncert}$	1.00000 mol/mol	$1.50 \cdot 10^{-3}$ mol/mol	normal	0.098	$150 \cdot 10^{-6}$ g/g	96.3 %
γ_U	0.097930 g/g	$150 \cdot 10^{-6}$ g/g				

Results:

Quantity	Value	Expanded Uncertainty	Coverage factor	Coverage
c_{238U}	$387.2 \cdot 10^{-6}$ mol/g	0.31 % (relative)	2.00	manual
c_U	$411.7 \cdot 10^{-6}$ mol/g	$1.3 \cdot 10^{-6}$ mol/g	2.00	manual
f_{234Ux}	$498.17 \cdot 10^{-6}$ mol/mol	$220 \cdot 10^{-9}$ mol/mol	2.00	manual
f_{235Ux}	0.058931 mol/mol	$18 \cdot 10^{-6}$ mol/mol	2.00	manual
f_{236Ux}	$62.556 \cdot 10^{-6}$ mol/mol	$25 \cdot 10^{-9}$ mol/mol	2.00	manual
f_{238Ux}	0.940508 mol/mol	$18 \cdot 10^{-6}$ mol/mol	2.00	manual
M_U	237.871466 g/mol	$53 \cdot 10^{-6}$ g/mol	2.00	manual
R_y	17.222 mol/mol	0.010 mol/mol	2.00	manual
γ_U	0.09793 g/g	$300 \cdot 10^{-6}$ g/g	2.00	manual



Annex 4. Calculation of isotope abundance fractions, isotope mass fractions and molar mass for IRMM-2030

Urenco S7

Measurements performed using TIMS/MTE, according to Method Working Instruction: IMS-JRC.G-C1.1-WIN-0059 v1.0

All results of mass spectrometric measurements for this request are stored in "U:\Nuclear Safeguards\Nuclear\TRITON DATA - SHARED\URENCO 2018". Filename: "EXTENDED T200717-23-27 MTE - Urenco S7 - F-D-00528 Revision 02.xlsm"

Model Equation:

{--- final ratios of sample---}

$$M_U = M_{233U} \cdot f_{233U} + M_{234U} \cdot f_{234U} + M_{235U} \cdot f_{235U} + M_{236U} \cdot f_{236U} + M_{238U} \cdot f_{238U};$$

$$f_{233U} = R_{233/238U}/\Sigma R_U;$$

$$f_{234U} = R_{234/238U}/\Sigma R_U;$$

$$f_{235U} = R_{235/238U}/\Sigma R_U;$$

$$f_{236U} = R_{236/238U}/\Sigma R_U;$$

$$f_{238U} = 1/\Sigma R_U;$$

$$\Sigma R_U = R_{233/238U} + R_{234/238U} + R_{235/238U} + R_{236/238U} + 1;$$

$$w_{233U} = f_{233U} \cdot M_{233U}/M_U;$$

$$w_{234U} = f_{234U} \cdot M_{234U}/M_U;$$

$$w_{235U} = f_{235U} \cdot M_{235U}/M_U;$$

$$w_{236U} = f_{236U} \cdot M_{236U}/M_U;$$

$$w_{238U} = f_{238U} \cdot M_{238U}/M_U;$$

List of Quantities:

Quantity	Unit	Definition
$R_{233/238U}$	mol/mol	isotope amount ratio n_{233}/n_{238} of U
$R_{234/238U}$	mol/mol	isotope amount ratio n_{234}/n_{238} of U
$R_{235/238U}$	mol/mol	isotope amount ratio n_{235}/n_{238} of U
$R_{236/238U}$	mol/mol	isotope amount ratio n_{236}/n_{238} of U
M_U	g/mol	molar mass of U
f_{233U}	mol/mol	isotope amount fraction of ^{233}U in U
f_{234U}	mol/mol	isotope amount fraction of ^{234}U in U
f_{235U}	mol/mol	isotope amount fraction of ^{235}U in U
f_{236U}	mol/mol	isotope amount fraction of ^{236}U in U
f_{238U}	mol/mol	isotope amount fraction of ^{238}U in U
w_{233U}	mol/mol	isotope mass fraction of ^{233}U in U
w_{234U}	mol/mol	isotope mass fraction of ^{234}U in U
w_{235U}	mol/mol	isotope mass fraction of ^{235}U in U
w_{236U}	mol/mol	isotope mass fraction of ^{236}U in U
w_{238U}	mol/mol	isotope mass fraction of ^{238}U in U

Quantity	Unit	Definition
M_{233U}	g/mol	atomic mass for ^{233}U
M_{234U}	g/mol	atomic mass for ^{234}U
M_{235U}	g/mol	atomic mass for ^{235}U
M_{236U}	g/mol	atomic mass for ^{236}U
M_{238U}	g/mol	atomic mass for ^{238}U
ΣR_U	mol/mol	sum of isotope ratios for U

$R_{233/238U}$:
Type B normal distribution
Value: 0 mol/mol
Expanded Uncertainty: 0 mol/mol
Coverage Factor: 2

$R_{234/238U}$:
Type B normal distribution
Value: 0.00052968 mol/mol
Expanded Uncertainty: 0.00000023 mol/mol
Coverage Factor: 2

$R_{235/238U}$:
Type B normal distribution
Value: 0.062659 mol/mol
Expanded Uncertainty: 0.000020 mol/mol
Coverage Factor: 2

$R_{236/238U}$:
Type B normal distribution
Value: 0.000066513 mol/mol
Expanded Uncertainty: 0.000000027 mol/mol
Coverage Factor: 2

M_{233U}:
Type B normal distribution
Value: 233.039627 g/mol
Expanded Uncertainty: 0.000003 g/mol
Coverage Factor: 1.0

The mass of the isotope is read from \\Sim_server\gumwork\wizards\ELTABLE.CSV.

M_{234U}:
Type B normal distribution
Value: 234.0409503 g/mol
Expanded Uncertainty: 0.0000024 g/mol
Coverage Factor: 2

Meng Wang et al (2021), The AME 2020 atomic mass evaluation, Chinese Phys. C 45 030003

M_{235U}:
Type B normal distribution
Value: 235.0439281 g/mol
Expanded Uncertainty: 0.0000024 g/mol
Coverage Factor: 2.0

Meng Wang et al (2021), The AME 2020 atomic mass evaluation, Chinese Phys. C 45 030003

M_{236U}:
Type B normal distribution
Value: 236.0455661 g/mol
Expanded Uncertainty: 0.0000024 g/mol
Coverage Factor: 2.0

Meng Wang et al (2021), The AME 2020 atomic mass evaluation, Chinese Phys. C 45 030003

M_{238U}: Type B normal distribution
Value: 238.0507869 g/mol
Expanded Uncertainty: 0.0000032 g/mol
Coverage Factor: 2.0

Meng Wang et al (2021), The AME 2020 atomic mass evaluation, Chinese Phys. C 45 030003

Interim Results:

Quantity	Value	Standard Uncertainty
ΣR_U	1.0632552 mol/mol	$10.0 \cdot 10^{-6}$ mol/mol

Results:

Quantity	Value	Expanded Uncertainty	Coverage factor	Coverage
M_U	237.871466 g/mol	$53 \cdot 10^{-6}$ g/mol	2.00	manual
f_{234U}	$498.17 \cdot 10^{-6}$ mol/mol	$220 \cdot 10^{-9}$ mol/mol	2.00	manual
f_{235U}	0.058931 mol/mol	$18 \cdot 10^{-6}$ mol/mol	2.00	manual
f_{236U}	$62.556 \cdot 10^{-6}$ mol/mol	$25 \cdot 10^{-9}$ mol/mol	2.00	manual
f_{238U}	0.940508 mol/mol	$18 \cdot 10^{-6}$ mol/mol	2.00	manual
w_{234U}	$490.15 \cdot 10^{-6}$ mol/mol	$210 \cdot 10^{-9}$ mol/mol	2.00	manual
w_{235U}	0.058231 mol/mol	$18 \cdot 10^{-6}$ mol/mol	2.00	manual
w_{236U}	$62.076 \cdot 10^{-6}$ mol/mol	$25 \cdot 10^{-9}$ mol/mol	2.00	manual
w_{238U}	0.941217 mol/mol	$17 \cdot 10^{-6}$ mol/mol	2.00	manual

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