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Preparation and Certification of IRMM-3636, IRMM-3636a and IRMM-3636b

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The mission of the IRMM is to promote a common and reliable European measurement system in support of EU policies.

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1. Abstract

Isotope reference materials IRMM-3636 was prepared by gravimetrically mixing of solutions from highly enriched 99.96% ^{233}U (IRMM-3630) and highly enriched 99.97% ^{236}U (IRMM-3660) in order to obtain an isotopic ratio $n(^{233}\text{U})/n(^{236}\text{U})$ close to unity. IRMM-3636a and IRMM-3636b were prepared from IRMM-3636 by gravimetrical dilution.

The certified isotope content values[1] for IRMM-3636 of $4.27682(54)\cdot10^{-6}$ mol U per g of solution, IRMM-3636a of $4.27988(54)\cdot10^{-7}$ mol U per g of solution and for IRMM-3636b of $2.21802(61)\cdot10^{-8}$ mol U per g of solution and the certified isotope amount ratios have been established by mass metrology, subsequently the primary solution IRMM-3636 was verified by isotope mass spectrometry. The methodology used in the preparation and certification was similar to that of comparable uranium mixtures made in the past [2] [3].

The uncertainties contributing to the final uncertainties of the isotopic ratios are the weighing errors, the measured impurities in each starting material, the stoichiometry of the oxides and the isotopic abundances of each of the starting materials. The method for the preparation and mixing is described and the certification and verification procedures are reported.

Verification of IRMM-3636 was performed by TRITON TIMS measurements using Faraday collectors and secondary electron multiplier in combination with an RPQ energy filter for improved abundance sensitivity. The results agreed well with the certified values obtained from the mixing calculations.

By mixing the $n(^{233}\text{U})/n(^{236}\text{U})$ double spike with a sample and applying internal mass fractionation correction, the $n(^{235}\text{U})/n(^{238}\text{U})$ ratio of a given sample can be determined with the highest possible accuracy.

The Isotopic Reference Materials IRMM-3636, IRMM-3636a and IRMM-3636b are part of a systematic IRMM programme to supply Isotopic Reference Materials of various isotopes at different concentrations The Isotopic Reference Material is supplied in a sealed quartz ampoule containing 1 mL of a 1 M nitric acid solution.

2. Introduction

In nature, the major uranium isotopes are ^{235}U and ^{238}U, the minor isotopes with a low isotopic abundance are ^{234}U and ^{236}U. The abundance of ^{236}U covers a very wide range from 10^{-2} in a nuclear reactor down to 10^{-11} in nature. The additional nuclear safeguards protocol at present in place in some countries allows for environmental sampling within nuclear sites. Small releases will occur in the environment from reprocessing or enrichment nuclear installation. Sampling environmental materials in the vicinity of such installations could be used to identify or detect undeclared nuclear activities, which could also provide a signature for a particular installation.

The measurement of the ^{236}U abundance proves to be a tool for tracing of sources of material and also provides information on the enrichment processes of uranium. This makes these measurements a valuable and important tool in nuclear safeguards. As a consequence there is a need for certified isotopic reference materials of uranium.

In the frame of the IRMM programme to supply Isotopic Reference Materials a series of uranium reference materials traceable to the SI has been prepared with the highest metrological quality for certified isotopic ratios and contents.

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[1] Note: 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 guide. 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.

In this report the preparation and certification of a so-called "$^{233}U/^{236}U$ Double Spike", a synthetic uranium isotope mixture produced by gravimetrical mixing of highly enriched 99.96% ^{233}U (IRMM-3630) and highly enriched 99.97% ^{236}U (IRMM-3660) with $n(^{233}U)/n(^{236}U)$ isotope ratio close to unity and the smallest possible combined uncertainty is described.

The Double Spike is characterized by a $n(^{233}U)/n(^{236}U)$ ratio of 1:1 and quite low abundances of ^{234}U, ^{235}U and ^{238}U (all <0.05%). The main advantage of this material is the possibility to perform an internal fractionation correction for measurements of uranium samples with low ^{236}U abundance, which applies for all natural samples. The use of the internal fractionation correction allows measurements of $n(^{235}U)/n(^{238}U)$ ratios as well as even $n(^{234}U)/n(^{238}U)$ ratios with significantly improved precision compared to external normalization using a (close to) natural isotope reference material.

Therefore the Double Spike is very useful for applications in various fields such as nuclear safeguards, environmental and geochemical research. In nuclear safeguards the $n(^{235}U)/n(^{238}U)$ and $n(^{234}U)/n(^{238}U)$ ratios have to be measured for isotopic fingerprinting and characterization of uranium ore materials [1].

Furthermore, $n(^{235}U)/n(^{238}U)$ ratios have to be measured as part of U-Pb-geochronology investigations. In particular, $n(^{234}U)/n(^{238}U)$ ratios are of interest in geochemical research for disequilibrium studies and U-Th-geochronology. By mixing the Double Spike with a ^{229}Th Spike, the determination of $n(^{230}Th)/n(^{238}U)$ activity ratios could be improved as well. According to the results of a recent customer survey the new Double Spike in two concentrations available, will be a very much appreciated new synthetic isotope reference material.

3. Design of IRMM-3636, IRMM-3636a and b

The primary solution IRMM-3636 is designed such that the material consists of equivalent parts of two individual isotopes $n(^{233}U)/n(^{236}U)$ with a ratio of one. Both starting materials, highly enriched 99.96% ^{233}U (IRMM-3630) and 99.97% ^{236}U (IRMM-3660), were selected to prepare the primary solution. A further 10-fold dilution of the primary mixture was made to prepare IRMM-3636a; a 200-fold dilution of the primary mixture was made to prepare IRMM-3636b

The starting solutions available and successfully used for the preparation of IRMM-074 and IRMM-075 have been used. The procedure for chemical purification of the uranium used in the preparation of both series as extensively described elsewhere [2] [3] was applied.

The isotopic composition of IRMM-3636 was calculated from the masses of the starting solutions mixed together and their respective isotopic compositions. Because of the very high enrichments of the starting materials, the contribution of the uncertainty from isotopic abundances to the combined uncertainty of the certified ratios and content of the mixture is small.

4. Certification of isotopic ratios in starting materials

The isotopic composition of the IRMM-3636 starting materials (enriched ^{233}U and ^{236}U materials) were measured using the (modified) total evaporation technique on the Triton TIMS [6][7]. By using a total evaporation technique the measurement is continued until the sample is exhausted. This is done in order to minimize mass fractionation effects for which nonetheless corrections were made.

The following measurements were made on the Triton TIMS:

- ^{233}U enriched material, IRMM-3630. Certified values are listed below in Table 1:

Table 1: Isotopic composition of highly enriched ^{233}U, Lot BC02153

Certified amount ratios	
$n(^{234}U)/n(^{233}U)$	0.000 359 21(47)
$n(^{235}U)/n(^{233}U)$	0.000 004 204 7(38)
$n(^{236}U)/n(^{233}U)$	0.000 000 025 36(19)
$n(^{238}U)/n(^{233}U)$	0.000 009 137(24)

amount fraction (·100)		mass fraction (·100)	
$n(^{233}U)/n(U)$	99.962 756(47)	$m(^{233}U)/m(U)$	99.962 578(47)
$n(^{234}U)/n(U)$	0.035 908(47)	$m(^{234}U)/m(U)$	0.036 062(47)
$n(^{235}U)/n(U)$	0.000 420 31(38)	$m(^{235}U)/m(U)$	0.000 423 92(38)
$n(^{236}U)/n(U)$	0.000 002 535(19)	$m(^{236}U)/m(U)$	0.000 002 568(19)
$n(^{238}U)/n(U)$	0.000 913 4(24)	$m(^{238}U)/m(U)$	0.000 933 0(24)

The molar mass of the uranium is 233.040 040 8(60) g·mol^{-1}

- ^{236}U enriched material, IRMM-3660. Certified values are listed below in Table 2.

Table 2: Isotopic composition of highly enriched ^{236}U, Lot BC02676

Certified amount ratios	
$n(^{233}U)/n(^{236}U)$	0.000 000 034 32(30)
$n(^{234}U)/n(^{236}U)$	0.000 000 001 222(82)
$n(^{235}U)/n(^{236}U)$	0.000 041 196(74)
$n(^{238}U)/n(^{236}U)$	0.000 225 50(38)

amount fraction (·100)		mass fraction (·100)	
$n(^{233}U)/n(U)$	0.000 003 431(30)	$m(^{233}U)/m(U)$	0.000 003 387(29)
$n(^{234}U)/n(U)$	0.000 000 122 2(82)	$m(^{234}U)/m(U)$	0.000 000 121 1(81)
$n(^{235}U)/n(U)$	0.004 118 5(74)	$m(^{235}U)/m(U)$	0.004 101 0(74)
$n(^{236}U)/n(U)$	99.973 334(38)	$m(^{236}U)/m(U)$	99.973 160(39)
$n(^{238}U)/n(U)$	0.022 544(38)	$m(^{238}U)/m(U)$	0.022 735(38)

The molar mass of the uranium is 236.045 971 7(43) g·mol^{-1}

5. Certification of IRMM-3636, IRMM3636a and b

Certified values for the isotope amount ratios, amount contents and uncertainties were calculated according to ISO/GUM recommendations [4] using the GUM Workbench [5].

The major contributors to the final uncertainties of the isotopic ratios and isotope amount content identified during the preparation of IRMM-3636 were the uncertainties from weighings of the mixture, uncertainties on certified values of the starting solutions of ^{233}U and ^{236}U and the dilution of the mixture. Components of uncertainty from the properties of the initial base materials (uranium oxides) such as chemical impurities, stoichiometry and of the certified isotope amount contents of the starting solutions are already accounted for in the certified isotope content of the starting solutions of ^{233}U (IRMM-3630) and ^{236}U (IRMM-3660).

The effect of the impurities and the stoichiometry in the starting solutions on the combined uncertainty of the certified ratios in the mixture was evaluated by designing a mathematical model in the GUM Workbench. In this mixing model the uncertainty on both values was put at 0.01% and a correlation factor of 0.8 was applied for both variables whereas the uncertainties for both, impurities and stoichiometry, in the models of both starting materials was set at 0 in order not have a double effect in the mixing.

Certified values for the isotope ratios and the uncertainty budget with the major components of uncertainty are listed in Table 3.

Table 3: Isotopic composition of IRMM-3636, IRMM-3636a and b

Certified amount ratios	
$n(^{233}U)/n(^{236}U)$	1.019 06(16)
$n(^{234}U)/n(^{236}U)$	0.000 366 06(48)
$n(^{235}U)/n(^{236}U)$	0.000 045 480(74)
$n(^{238}U)/n(^{236}U)$	0.000 234 81(38)

amount fraction (·100)		mass fraction (·100)	
$n(^{233}U)/n(U)$	50.455 8(39)	$m(^{233}U)/m(U)$	50.135 5(39)
$n(^{234}U)/n(U)$	0.018 125(24)	$m(^{234}U)/m(U)$	0.018 087(24)
$n(^{235}U)/n(U)$	0.002 251 8(37)	$m(^{235}U)/m(U)$	0.002 256 8(37)
$n(^{236}U)/n(U)$	49.512 2(39)	$m(^{236}U)/m(U)$	49.832 4(39)
$n(^{238}U)/n(U)$	0.011 626(19)	$m(^{238}U)/m(U)$	0.011 801(19)

The molar mass of the uranium is 234.528 74(12) g·mol^{-1}

Table 4: Uncertainty budget for certified $n(^{233}U)/n(^{236}U)$ in IRMM-3636

Quantity	Description	Value	%
$C_{U\text{-}in\text{-}3630}$	U amount content SM233	4.31346(50) µmol/g	54.3
$C_{U\text{-}in\text{-}3660}$	U amount content SM236	4.23157(22) µmol/g	10.4
m_{3630}	mass SM233 in mixture	60.0560(23) g	6.2
m_{3660}	mass SM236 in mixture	60.0670(23) g	6.2
δ_{233}	Impurities of uranium in SM233	0.00000(12)	11.2
δ_{236}	Impurities of uranium in SM236	0.00000(12)	11.2
f_{O233}	Stoichiometry of U_3O_8 in SM233	8.00000(92)	0.3
f_{O236}	Stoichiometry of U_3O_8 in SM236	8.00000(92)	0.3

The certified values for isotope contents and uncertainty budgets with the major components of uncertainty are listed in Table 5 and Table 6.

Table 5: Certified values for isotope content of IRMM-3636, 3636a and b

Quantity	Value	
IRMM-3636		
U amount content($C_{U\text{-}3636}$):	4.27682(54)	µmol U/g
U mass content ($\gamma_{U\text{-}3636}$):	1.00304(13)	mg U/g
^{236}U isotope amount content ($C_{236U\text{-}3636}$):	2.11755(26)	µmol ^{236}U/g
^{233}U isotope amount content ($C_{233U\text{-}3636}$):	2.15791(35)	µmol ^{233}U/g
^{236}U mass amount content ($\gamma_{236U\text{-}3636}$):	496.626(62)	µg^{236}U /g
^{233}U mass amount content ($\gamma_{233U\text{-}3636}$):	506.091(81)	µg^{233}U /g
IRMM-3636a		
U amount content($C_{U\text{-}3636a}$):	0.427988(54)	µmol U/g
U mass content ($\gamma_{U\text{-}3636a}$):	100.375(11)	µg U/g
^{236}U isotope amount content ($C_{236U\text{-}3636a}$):	0.211906(26)	µmol ^{236}U/g
^{233}U isotope amount content ($C_{233U\text{-}3636a}$):	0.215945(35)	µmol ^{233}U/g
^{236}U mass amount content ($\gamma_{236U\text{-}3636a}$):	49.6981(61)	µg^{236}U /g
^{233}U mass amount content ($\gamma_{233U\text{-}3636a}$):	50.6452(81)	µg^{233}U /g
IRMM-3636b		
U amount content($C_{U\text{-}3636b}$):	22.1802(61)	nmol U/g
U mass content ($\gamma_{U\text{-}3636b}$):	5.2019(14)	µg U/g
^{236}U isotope amount content ($C_{236U\text{-}3636b}$):	10.9819(30)	nmol ^{236}U/g
^{233}U isotope amount content ($C_{233U\text{-}3636b}$):	11.1912(33)	nmol ^{233}U/g
^{236}U mass amount content ($\gamma_{236U\text{-}3636b}$):	2.57557(71)	µg^{236}U /g
^{233}U mass amount content ($\gamma_{233U\text{-}3636b}$):	2.62466(77)	µg^{233}U /g

Table 6: Uncertainty budget for certified uranium amount contents of IRMM-3636, 3636a and b

Quantity	Description	Value		%
IRMM-3636				
$C_{U\text{-}in\text{-}3630}$	U amount content SM233	4.31346(50)	µmol/g	20.6
$C_{U\text{-}in\text{-}3660}$	U amount content SM236	4.23157(22)	µmol/g	3.8
m_{3630}	mass SM233 in mixture	60.0560(23)	g	2.4
m_{3660}	mass SM236 in mixture	60.0670(23)	g	2.3
evaporation correction	sampling from IRMM-3636a	-		70.9

Quantity	Description	Value	%
IRMM-3636a			
$c_{U\text{-}in\text{-}3630}$	U amount content SM233	4.31346(50) µmol/g	20.7
$c_{U\text{-}in\text{-}3660}$	U amount content SM236	4.23157(22) µmol/g	3.8
m_{3630}	mass SM233 in mixture	60.0560(23) g	2.4
m_{3660}	mass SM236 in mixture	60.0670(23) g	2.3
evaporation correction	sampling from IRMM-3636a	-	69.5
$m_{3636\text{-}for\text{-}3636a}$	mass IRMM-3636 for dilution	20.043900(23) g	0.8
$m_{3636a\text{-}total}$	total IRMM-3636a	200.24890(19) g	0.5
IRMM-3636b			
$c_{U\text{-}in\text{-}3630}$	U amount content SM233	4.31346(50) µmol/g	4.4
$c_{U\text{-}in\text{-}3660}$	U amount content SM236	4.23157(22) µmol/g	0.8
m_{3630}	mass SM233 in mixture	60.0560(23) g	0.5
m_{3660}	mass SM236 in mixture	60.0670(23) g	0.5
evaporation correction	sampling from IRMM-3636a	-	14.5
$m_{3636\text{-}for\text{-}3636a}$	mass IRMM-3636 for dilution	20.043900(23) g	79.1
$m_{3636a\text{-}total}$	total IRMM-3636a	200.24890(19) g	0.2

6. Ampouling of IRMM-3636, 3636a and b

Ampuling was carried out in a double section fume hood in the controlled area. The ampoules were filled with 1 mL of solution by means of a 5 mL size dispenser. Each ampoule contained respectively about 1 mg (IRMM-3636), 100 µg (IRMM-3636a) and 5 µg (IRMM-3636b) total uranium.

The fume hood was fitted with a new plastic interior. In the left part of the fume hood the top part was not covered since it would get too hot when the ampoules were sealed. In the right part the burner for the flame sealing of the ampoules (acetylene/oxygen flame) was installed, surrounded by fireproof plates. The left part was used to set up flask and liquid dispenser. A sufficient number of clean ampoules were brought into the controlled area from the clean lab, as well as dispensers and tubing. The area around and under the filling station was covered with a fresh layer of clean room wipes prior to ampuling.

The flask containing the Double Spike solution to be processed was then opened. The dispenser was carefully fitted onto the flask, taking care to keep the ends of the tubing clean. One tube was then carefully inserted into the flask so that it reached the bottom of the flask. The other tubing was inserted into the ampoule. The required volume was then transferred from flask into ampoule with the dispenser. The ampoule was inspected that there was no solution in the neck and placed into a rack.

From there it was put into a small PTFE holder and sealed using an oxygen-acetylene flame. After visual inspection the ampoule was placed into a rack to cool. This was done in a continuous process, with one ampoule being processed in less than a minute, on the average. The sequence of filling is shown in Table 7 below.

The same procedure was applied for the ampuling of both IRMM-3636a and IRMM-3636b

From earlier experiments and testing carried out during similar operations [8], the maximum possible contamination from environmental uranium during the preparation of the mother solutions, dilutions, ampoule filling and the sealing process is estimated to be about 26 pg uranium ($1.1 \cdot 10^{-6}$ mol). Possible contamination at this level of uranium with natural isotopic composition has no significant effect on the isotopic ratios of IRMM-3636, 3636a or 3636b solutions.

Table 7: Sequence of ampoule filling for IRMM-3636, 3636a and b

	Date	Number of ampoules sealed
3636	10-06-2008	80
3636a	18-03-2008	196
3636b	17-03-2008	95

7. Verification of certified isotope amount ratios

The certified isotope ratio $n(^{233}U)/n(^{236}U)$ ratio was verified by isotopic measurements using a Thermo-Electron Triton TIMS at IRMM. For this measurement a multi-dynamic technique was used in order to minimize any influence and uncertainty contributions arising from the Faraday amplifier gains and cup efficiencies. The uncertainties for the amplifier gains were found to be at the level of 20 ppm by repeated measurements of the amplifier baselines. For the relative cup efficiencies a mutual agreement at the level of < 5 ppm was shown.

The IRMM-3636 "sample" was mixed with an approximate amount ratio of 1:1 with the synthetic isotope reference material IRMM-3050. IRMM-3050 is an original synthetic mixture of highly enriched ^{235}U (IRMM-3650) and ^{238}U (IRMM-3680) isotopic reference material primary solutions, part of which was mixed with a ^{233}U isotopic reference material, primary solution, to obtain the IRMM-074 series.

For the verification measurement the known $n(^{235}U)/n(^{238}U)$ ratio of 1.000259(81) in IRMM-3050 was used for internal mass fractionation correction to measure $n(^{233}U)/n(^{236}U)$ of the "sample" IRMM-3636. The very low uncertainty of the $n(^{235}U)/n(^{238}U)$ ratio for IRMM-074 are a result of mixing the purified oxides as solids in a flask and subsequently dissolivng to form a master solution. For the double spike, IRMM-3636, the primary solutions of ^{233}U and ^{236}U were mixed together which is reflected in a higher certified uncertainty.

For the verification measurement of IRMM-3636 a multi-dynamic procedure was applied in order to minimize any influence and uncertainty contributions arising from the Faraday amplifier gains and cup efficiencies. This was achieved by measuring the $n(^{235}U)/n(^{238}U)$ ratio of IRMM-3050 using the same pair of Faraday cups as the $n(^{233}U)/n(^{236}U)$ ratio of IRMM-3636. The mass cycle was arranged as follows:

Table 8: Mass cycle for multi-dynamic measurement technique

Step	Cup L3	Cup L1	Cup C	Cup H2	Integration time (s)	Idle time (s)
1	233U	**235U**	236U	**238U**	32	10
2	232.7	234.7	235.7	237.7	16	10
3	233.4	235.4	236.4	238.4	16	10
4		**233U**		**236U**	32	10

5		232.7		235.7	16	10
6		233.4		236.4	16	10

Lines 2, 3 and 5, 6 are introduced for background measurements close to the measured uranium peaks. Due to the multi-dynamic measurement technique the $n(^{235}U)/n(^{238}U)$ ratio of IRMM-3050 is measured using the same pair of Faraday cups (L1 and H2) as the $n(^{233}U)/n(^{236}U)$ ratio of IRMM-3636, emphasized in Table 8 by the bold mass numbers for ^{235}U and ^{238}U in step1 and for ^{233}U and ^{236}U in step 4.

As shown below in Table 9, this leads to a cancellation of all Faraday amplifier gains and cup efficiencies from the uncertain budget for the multi-dynamic measurement technique, but not for the static technique (only consisting of step1-3 of the mass cycle).

Within the multi-dynamic cycle the ^{233}U is also measured in Cup L3 (step1) in order to allow a (small) correction for the "minor" ^{235}U and ^{238}U abundances from IRMM-3636. These can be calculated using the known isotopic compositions of the ^{233}U and ^{236}U starting materials.

Table 9: Uncertainty budgets of IRMM-3636 (multi-dynamic and static measurement techniques)

Quantity	Multi-Dynamic (%)	Static (%)
Correction for ^{238}U in IRMM-3636	1.3	0.7
Correction for ^{235}U in IRMM-3636	0.2	0.1
$n(^{235}U)/n(^{238}U)$ measurement	5.4	2.3
$n(^{233}U)/n(^{236}U)$ measurement	5.4	2.3
Faraday Cups (gain, efficiency)		57.2
$n(^{235}U)/n(^{238}U)$ from IRMM-3050	88.1	37.6
Rel. Unc. of $n(^{233}U)/n(^{236}U)$ result:	0.0086	0.013

The result for the mass spectrometric verification measurement of IRMM-3636 is given by a ratio of $n(^{233}U)/n(^{236}U) = 1.019090(86)$. The relative uncertainty 0.0086% is only slightly higher than the relative uncertainty of 0.0081% for the IRMM-3050 synthetic reference material. This mass spectrometric result is in excellent agreement with the calculated ratio from the gravimetric mixing, which is $n(^{233}U)/n(^{236}U) = 1.01906(16)$, see Figure 1.

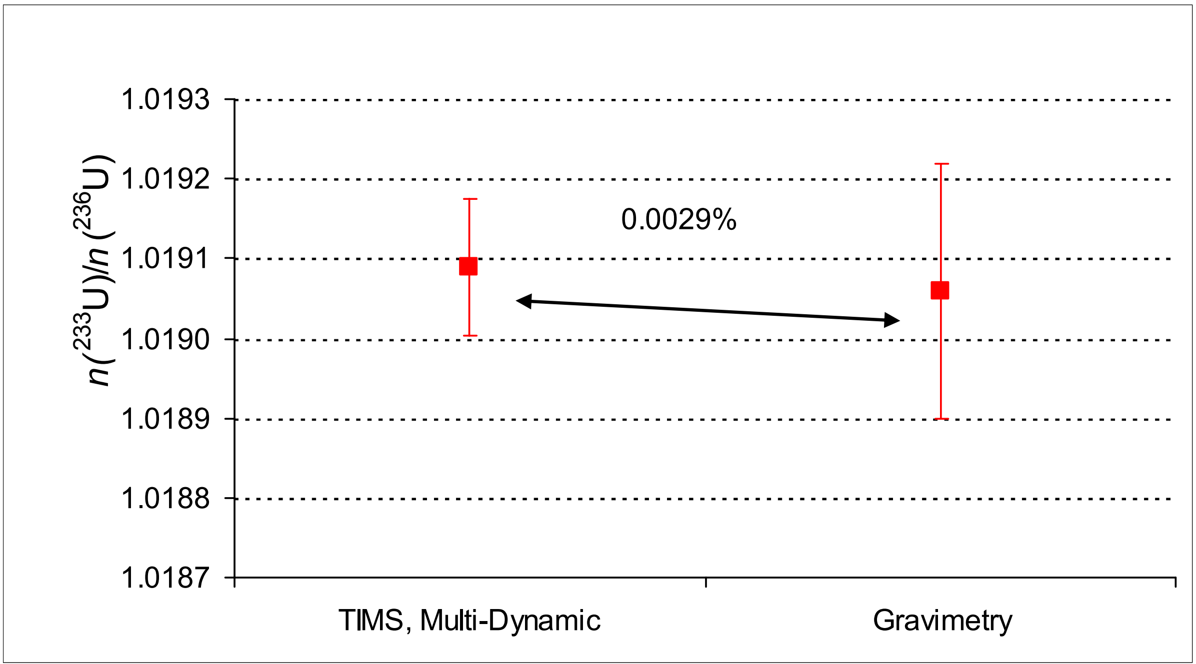

Figure 1: Verification measurement of isotope ratios in IRMM-3636

Isotope reference materials such as the double spike IRMM-3636 are prepared to be used as standards in isotope mass spectrometry. In order to avoid circular reasoning the certified $n(^{233}U)/n(^{236}U)$ ratio of this reference material should be as independent as possible from mass spectrometric measurements. Therefore the *gravimetrically* determined ratio and its uncertainty are used for the certification of the double spike IRMM-3636. As a result from the mass spectrometric verification measurement, the very small and insignificant relative difference of 0.0029% between the measured and certified ratios provides a strong confidence to the certified $n(^{233}U)/n(^{236}U)$ ratio of IRMM-3636. This excellent agreement also confirms the assumption of achieving similar impurity levels and stoichiometry for all enriched starting materials of ^{233}U, ^{235}U, ^{236}U and ^{238}U prior to the preparation of the mixtures.

Because of the high enrichment of the starting materials (99.96% for ^{233}U in IRMM-3630 and 99.97% for ^{236}U in IRMM-3660) the calculated gravimetric mixing proportions are confirmed by the verification of the "major" $n(^{233}U)/n(^{236}U)$ isotope ratio. All further isotopic ratios and isotopic abundances of the "minor" isotopes ^{234}U, ^{235}U and ^{238}U in IRMM-3636 are therefore calculated using the confirmed mixing proportions and the measured abundances of ^{234}U, ^{235}U and ^{238}U in the starting materials IRMM-3630 and IRMM-3660

8. Verification of uranium amount concentration of IRMM-3636

The verification of the uranium amount content of IRMM-3636 and therefore also for IRMM-3636a and b was accomplished by IDMS using the ^{233}U spike IRMM-040a and ^{238}U spike IRMM-052. Since the $n(^{233}U)/n(^{236}U)$ isotope ratio was already verified, the determination of the ^{236}U concentration was basically sufficient. The results are also shown in Figure 2. The verification of the U amount content of IRMM-3636 can therefore be considered successful.

a) IDMS using the ^{233}U spike IRMM-040a

Three aliquots from IRMM-3636 were weighed, transferred into a recipient and mixed with a weighed amount the ^{233}U spike IRMM-040a. The certificate for mass metrology is shown in Annex. Using the simplified IDMS equation, the ^{236}U concentrations were calculated for the 3 mixtures, the final results are presented in the Table 10. As a conclusion, the total uranium concentration C(U) of IRMM-3636, calculated from the gravimetrical data, was confirmed by IDMS measurements within uncertainties. Using the simplified IDMS equation, the ^{236}U concentrations were calculated for the 3 mixtures, the final results are presented in the Table 10. The total uranium concentration C(U) of IRMM-3636, calculated from the gravimetrical data, was confirmed by IDMS measurements without significant difference.

Table 10: Results from IDMS measurements for IRMM-3636 using the ^{233}U spike IRMM-040a.

Average C(^{236}U) from IDMS, measured	0.00000211500 mol/g
SD	0.00000000077 mol/g
RSD	0.042%
Rel Uc k=2 (main contribution is from IRMM-40a)	0.18%
Gravimetric C(^{236}U)	0.0000021162 mol/g
Rel. Deviation measured versus gravimetric result:	-0.054%
Rel Uc k=2, of the deviation	0.18%

b) IDMS using the ^{238}U spike IRMM-052

IRMM-3636 was mixed with the ^{238}U spike IRMM-052 in 6 different proportions, using 3 different ampoules of IRMM-052 Mass Metrology data are given on certificate in annex. Using the simplified IDMS equation, the ^{236}U concentrations were calculated for the 6 blends, the final results are presented in Table 11. The total uranium concentration C(U) of IRMM-3636, calculated from the gravimetrical data, was confirmed by IDMS measurements without significant difference.

Table 11: Results from IDMS measurements for IRMM-3636 using the ^{238}U spike IRMM-052

Average C(^{236}U) from IDMS, measured	0.0000021163 mol/g
SD	0.0000000011 mol/g
Rel Uc k=2 (main contribution is from IRMM-40a)	0.054%
Gravimetric C(^{236}U)	0.00000211755 mol/g
Rel. Deviation measured versus gravimetric result:	-0.060%
Rel Uc k=2, of the deviation	0.056%

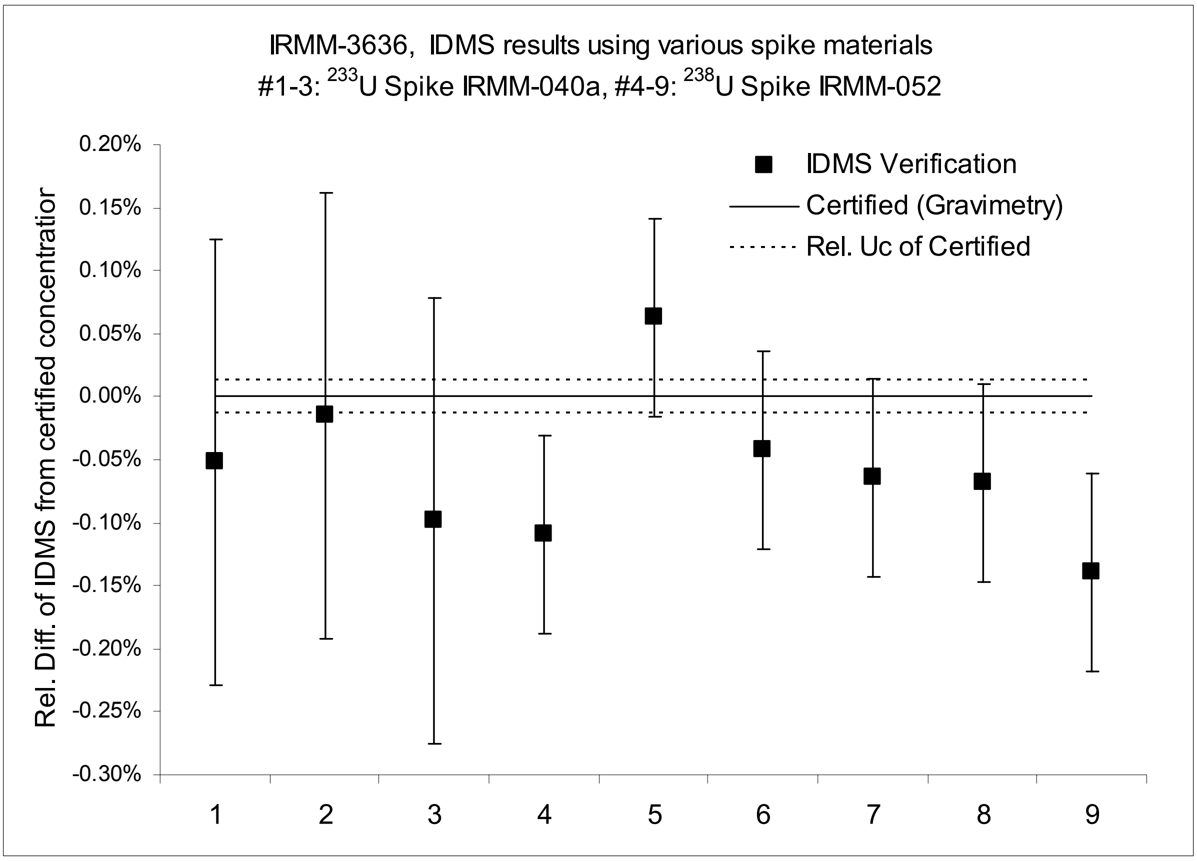

Figure 2: Verification measurements of uranium isotope content in IRMM-3636

9. Conclusions

The methodology and techniques used in the preparation of synthetic mixtures IRMM-074 and IRMM-075 have again been applied successfully for the preparation of the double spike IRMM-3636 and of its dilutions IRMM-3636a and b.

The series have been prepared and certified values of the isotope amount ratios and contents have been calculated based on the weights of oxides and solutions and verified by the uncertainties of the mixing calculations and by independent TIMS measurements.

The isotopic reference material IRMM-3636a and b are commercially available from IRMM; because of the very small amount of material available the primary solution IRMM-3636 will only be used for internal purposes and not be available externally.

10. References

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Figure 3: Certificate IRMM-3636

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Isotope Measurements (Geel)

CERTIFICATE
SPIKE ISOTOPIC REFERENCE MATERIAL IRMM-3636

2.117 55(26) · 10^{-6} mol (^{236}U) · g^{-1} (solution)

The Spike Isotopic Reference Material is supplied with an isotope amount content of ^{236}U certified as above.

The amounts of other uranium isotopes present are related to the ^{236}U content through the following certified amount ratios:

$n(^{233}U)/n(^{236}U)$:	1.019 06(16)
$n(^{234}U)/n(^{236}U)$:	0.000 366 06(48)
$n(^{235}U)/n(^{236}U)$:	0.000 045 480(74)
$n(^{238}U)/n(^{236}U)$:	0.000 234 81(38)

This corresponds to an isotopic composition with the following abundances:

	amount fraction (·100)		mass fraction (·100)
$n(^{233}U)/n(U)$	50.455 8(39)	$m(^{233}U)/m(U)$	50.135 5(39)
$n(^{234}U)/n(U)$	0.018 125(24)	$m(^{234}U)/m(U)$	0.018 087(24)
$n(^{235}U)/n(U)$	0.002 251 8(37)	$m(^{235}U)/m(U)$	0.002 256 8(37)
$n(^{236}U)/n(U)$	49.512 2(39)	$m(^{236}U)/m(U)$	49.832 4(39)
$n(^{238}U)/n(U)$	0.011 626(19)	$m(^{238}U)/m(U)$	0.011 801(19)

The molar mass of the uranium in this sample is 234.528 74(12) g·mol^{-1}

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

4.276 82(54) · 10^{-6}	mol (U) · g^{-1} (solution)
2.157 91(35) · 10^{-6}	mol (^{233}U) · g^{-1} (solution)
1.003 04(13) · 10^{-3}	g (U) · g^{-1} (solution)
0.499 838(62) · 10^{-3}	g (^{236}U) · g^{-1} (solution)
0.502 878(81) · 10^{-3}	g (^{233}U) · g^{-1} (solution)

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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 this Isotopic Reference Material will, therefore, also be traceable to the SI unit system.

2. All uncertainties indicated are expanded uncertainties $U = k \cdot u_c$ where u_c is the combined standard uncertainty estimated following ISO/GUM recommendations[1]. 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.

3. This Reference Material was prepared by metrological weighing of highly enriched uranium base materials and dissolution in HNO_3. Subsequently the solution was dispensed into individual units.

4. Values for molar isotope abundance ratios are valid for 1 July 2007.

5. The Isotopic Reference Material IRMM-3636 comes in a flame-sealed quartz ampoule containing about 4.3 µmol uranium in about 1 mL of a chemically stable 1 M nitric acid solution.

6. The atomic masses, used in the calculations, are[2]

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

7. 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.

8. Full details on the certification procedure can be found in the Certification Report EUR 23408 EN [3]

Chemical purification of the $^{236}U_3O_8$ and $^{233}U_3O_8$ starting materials was performed by R Eykens and F Kehoe.

Weighing and preparation of the Isotopic Reference Material was performed by R Eykens. The ampoulation of this Isotopic Reference Material was accomplished by S Werelds, M Peeters, R Eykens and A Verbruggen.

Characterization of the enriched isotopes from which IRMM-3636 was prepared and verification measurements were performed by S Richter and H Kühn on samples prepared by F Kehoe and A Alonso Muñoz.

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[1] International Organisation for Standardisation, Guide to the expression of Uncertainty in Measurement, ©ISO, ISBN 92-67-10188-9, Geneva, Switzerland, 1993

[2] G. Audi and A.H. Wapstra, The 2003 atomic mass evaluation, Nucl Phys A729(2003) 337-676.

[3] A. Verbruggen, A. Alonso, R. Eykens, F. Kehoe, H. Kühn, S. Richter, Y. Aregbe, Preparation and certification of IRMM-3636 and 3636a, Report EUR 23408 EN

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	P Taylor	Y Aregbe
June 2008	Head Isotope Measurements Unit	IRMM Safeguards Coordinator

Figure 4: Certificate IRMM-3636a

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Isotope Measurements (Geel)

CERTIFICATE
SPIKE ISOTOPIC REFERENCE MATERIAL IRMM-3636a

2.119 06(26) · 10^{-7} mol (^{236}U) · g^{-1} (solution)

The Spike Isotopic Reference Material is supplied with an isotope amount content of ^{236}U certified as above.

The amounts of other uranium isotopes present are related to the ^{236}U content through the following certified amount ratios:

$n(^{233}U)/n(^{236}U)$:	1.019 06(16)
$n(^{234}U)/n(^{236}U)$:	0.000 366 06(48)
$n(^{235}U)/n(^{236}U)$:	0.000 045 480(74)
$n(^{238}U)/n(^{236}U)$:	0.000 234 81(38)

This corresponds to an isotopic composition with the following abundances:

amount fraction (·100)		mass fraction (·100)	
$n(^{233}U)/n(U)$	50.455 8(39)	$m(^{233}U)/m(U)$	50.135 5(39)
$n(^{234}U)/n(U)$	0.018 125(24)	$m(^{234}U)/m(U)$	0.018 087(24)
$n(^{235}U)/n(U)$	0.002 251 8(37)	$m(^{235}U)/m(U)$	0.002 256 8(37)
$n(^{236}U)/n(U)$	49.512 2(39)	$m(^{236}U)/m(U)$	49.832 4(39)
$n(^{238}U)/n(U)$	0.011 626(19)	$m(^{238}U)/m(U)$	0.011 801(19)

The molar mass of the uranium in this sample is 234.528 74(12) g·mol^{-1}

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

4.279 88(54) · 10^{-7}	mol (U) · g^{-1} (solution)
2.159 45(35) · 10^{-7}	mol (^{233}U) · g^{-1} (solution)
1.003 75(13) · 10^{-4}	g (U) · g^{-1} (solution)
0.500 195(62) · 10^{-5}	g (^{236}U) · g^{-1} (solution)
0.503 237(81) · 10^{-5}	g (^{233}U) · g^{-1} (solution)

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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 this Isotopic Reference Material will, therefore, also be traceable to the SI unit system.

2. All uncertainties indicated are expanded uncertainties $U = k \cdot u_c$ where u_c is the combined standard uncertainty estimated following ISO/GUM recommendations[1]. 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.

3. This Reference Material was prepared by metrological dilution of IRMM-3636 which was prepared by metrological weighing of high enriched uranium base materials. and dissolution in HNO_3. Subsequently the diluted solution was dispensed into individual units.

4. Values for molar isotope abundance ratios are valid for 1 July 2007.

5. The Isotopic Reference Material IRMM-3636a comes in a flame-sealed quartz ampoule containing about 0.42 µmol uranium in about 1 mL of a chemically stable 1 M nitric acid solution.

6. The atomic masses, used in the calculations, are[2]

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

7. 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.

8. Full details on the certification procedure can be found in the Certification Report EUR 23408 EN [3]

Chemical purification of the $^{236}U_3O_8$ and $^{233}U_3O_8$ starting materials was performed by R Eykens and F Kehoe.

Weighing and preparation of the Isotopic Reference Material was performed by R Eykens. The ampoulation of this Isotopic Reference Material was accomplished by S Werelds, M Peeters, R Eykens and A Verbruggen.

[1] International Organisation for Standardisation, Guide to the expression of Uncertainty in Measurement, ©ISO, ISBN 92-67-10188-9, Geneva, Switzerland, 1993
[2] G. Audi and A.H. Wapstra, The 2003 atomic mass evaluation, Nucl Phys A729(2003) 337-676.
[3] A. Verbruggen, A. Alonso, R. Eykens, F. Kehoe, H. Kühn, S. Richter, Y. Aregbe, Preparation and certification of IRMM-3636 and 3636a, Report EUR 23408 EN

Characterization of the enriched isotopes from which IRMM-3636 was prepared and verification measurements were performed by S Richter and H Kühn on samples prepared by F Kehoe and A Alonso Muñoz.

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	P Taylor	Y Aregbe
May 2008	Head Isotope Measurements Unit	IRMM Safeguards Coordinator

Figure 5: Certificate IRMM-3636b

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Institute for reference materials and measurements
Isotope Measurements (Geel)

CERTIFICATE
SPIKE ISOTOPIC REFERENCE MATERIAL IRMM-3636b

1.098 19(30) · 10^{-8} mol (^{236}U) · g^{-1} (solution)

The Spike Isotopic Reference Material is supplied with an isotope amount content of ^{236}U certified as above.

The amount of other uranium isotopes present are related to the ^{236}U content through the following certified amount ratios:

$n(^{233}\mathrm{U})/n(^{236}\mathrm{U})$:	1.019 06(16)
$n(^{234}\mathrm{U})/n(^{236}\mathrm{U})$:	0.000 366 06(48)
$n(^{235}\mathrm{U})/n(^{236}\mathrm{U})$:	0.000 045 480(74)
$n(^{238}\mathrm{U})/n(^{236}\mathrm{U})$:	0.000 234 81(38)

This corresponds to an isotopic composition with the following abundances:

amount fraction (·100)		mass fraction (·100)	
$n(^{233}\mathrm{U})/n(\mathrm{U})$	50.455 8(39)	$m(^{233}\mathrm{U})/m(\mathrm{U})$	50.135 5(39)
$n(^{234}\mathrm{U})/n(\mathrm{U})$	0.018 125(24)	$m(^{234}\mathrm{U})/m(\mathrm{U})$	0.018 087(24)
$n(^{235}\mathrm{U})/n(\mathrm{U})$	0.002 251 8(37)	$m(^{235}\mathrm{U})/m(\mathrm{U})$	0.002 256 8(37)
$n(^{236}\mathrm{U})/n(\mathrm{U})$	49.512 2(39)	$m(^{236}\mathrm{U})/m(\mathrm{U})$	49.832 4(39)
$n(^{238}\mathrm{U})/n(\mathrm{U})$	0.011 626(19)	$m(^{238}\mathrm{U})/m(\mathrm{U})$	0.011 801(19)

The molar mass of the uranium in this sample is 234.528 74(12) g·mol^{-1}

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

2.218 02(61) · 10^{-8}	mol (U) · g^{-1} (solution)
1.119 12(33) · 10^{-8}	mol (^{233}U) · g^{-1} (solution)
5.201 9(14) · 10^{-6}	g (U) · g^{-1} (solution)
2.592 23(71) · 10^{-6}	g (^{236}U) · g^{-1} (solution)
2.607 99(76) · 10^{-6}	g (^{233}U) · g^{-1} (solution)

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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 this Isotopic Reference Material will, therefore, also be traceable to the SI unit system.

2. All uncertainties indicated are expanded uncertainties $U = k \cdot u_c$ where u_c is the combined standard uncertainty estimated following ISO/GUM recommendations[1]. 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.

3. This Reference Material was prepared by metrological dilution of IRMM-3636 which was prepared by metrological weighing of high enriched uranium base materials. and dissolution in HNO_3. Subsequently the diluted solution was dispensed into individual units.

4. Values for molar isotope abundance ratios are valid for 1 July 2007.

5. The Isotopic Reference Material IRMM-3636b comes in a flame-sealed quartz ampoule containing about 22 nmol uranium in about 1 mL of a chemically stable 1 M nitric acid solution.

6. The atomic masses, used in the calculations, are[2]

^{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}

7. 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.

8. Full details on the certification procedure can be found in the Certification Report EUR 23408 EN [3]

Chemical purification of the $^{236}U_3O_8$ and $^{233}U_3O_8$ starting materials was performed by R Eykens and F Kehoe.

Weighing and preparation of the Isotopic Reference Material was performed by R Eykens. The ampoulation of this Isotopic Reference Material was accomplished by S Werelds, M Peeters, R Eykens and A Verbruggen.

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[1] International Organisation for Standardisation, Guide to the expression of Uncertainty in Measurement, ©ISO, ISBN 92-67-10188-9, Geneva, Switzerland, 1993
[2] G. Audi and A.H. Wapstra, The 2003 atomic mass evaluation, Nucl Phys A729(2003) 337-676.
[3] A. Verbruggen, A. Alonso, R. Eykens, F. Kehoe, H. Kühn, S. Richter, Y. Aregbe, Preparation and certification of IRMM-3636 and 3636a, Report EUR 23408 EN

Characterization of the enriched isotopes from which IRMM-3636 was prepared and verification measurements were performed by S Richter and H Kühn on samples prepared by F Kehoe and A Alonso Muñoz.

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 May 2008	P Taylor Head Isotope Measurements Unit	Y Aregbe IRMM Safeguards Coordinator

European Commission

EUR 23408 EN – Joint Research Centre – Institute for Reference Materials and Measurements

Title: Preparation and Certification Report IRMM-3636, IRMM-3636a and IRMM-3636b

Author(s): A. Verbruggen, R. Eykens, F. Kehoe, H. Kühn , S. Richter,Y. Aregbe

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Abstract

Isotope reference materials IRMM-3636 was prepared by gravimetrically mixing of solutions from highly enriched 99.96% ^{233}U (IRMM-3630) and highly enriched 99.97% ^{236}U (IRMM-3660) in order to obtain an isotopic ratio $n(^{233}U)/n(^{236}U)$ close to unity. IRMM-3636a and IRMM-3636b were prepared from IRMM-3636 by gravimetrical dilution. The primary solution, IRMM-3636 is for the time being not yet available for external users.

The certified isotope content values for IRMM-3636a of $4.27988(54)\cdot10^{-7}$ mol U per g of solution and for IRMM-3636b of $2.21802(61)\cdot10^{-8}$ mol U per g of solution and the certified isotope amount ratios have been established by mass metrology, subsequently the primary solution IRMM-3636 was verified by isotope mass spectrometry. The methodology used in the preparation and certification was similar to that of comparable uranium mixtures made in the past.

The uncertainties contributing to the final uncertainties of the isotopic ratios are the weighing errors, the measured impurities in each starting material, the stoichiometry of the oxides and the isotopic abundances of each of the starting materials. The method for the preparation and mixing is described and the certification and verification procedures are reported.

Verification of IRMM-3636 was performed by TRITON TIMS measurements using Faraday collectors and secondary electron multiplier in combination with an RPQ energy filter for improved abundance sensitivity. The results agreed well with the certified values obtained from the mixing calculations.

By mixing the $n(^{233}U)/n(^{236}U)$ double spike with a sample and applying internal mass fractionation correction, the $n(^{235}U)/n(^{238}U)$ ratio of a given sample can be determined with the highest possible accuracy.

The Isotopic Reference Materials IRMM-3636a and IRMM-3636b are part of a systematic IRMM programme to supply Isotopic Reference Materials of various isotopes at different concentrations The Isotopic Reference Material is supplied in a sealed quartz ampoule containing 1 mL of a 1 M nitric acid solution.

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