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Preparation and Certification of IRMM-3660, IRMM-3660a and IRMM-3660b

A. Verbruggen, A. Alonso, R. Eykens, F. Kehoe,
H. Kühn, S. Richter, Y. Aregbe

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Contact information
Address: Retieseweg 111, B2440 Geel, Belgium
E-mail: andre.verbruggen@ec.europa.eu
Tel.: +32 14 571 617
Fax: +32 14 571 863

http://irmm.jrc.ec.europa.eu/
http://www.jrc.ec.europa.eu/

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Table of Contents

1. Abstract

Isotope reference materials IRMM-3660 was prepared by dissolution of highly enriched 99.97% ^{236}U in nitric acid. IRMM-3660a and IRMM-3660b were prepared from IRMM-3660 by gravimetrical dilution.

The certified isotope content values[1] for IRMM-3660 of $4.23651(43)\cdot10^{-6}$ mol U per g of solution, IRMM-3660a of $4.22498(44)\cdot10^{-7}$ mol U per g of solution and for IRMM-3660b of $4.2655(11)\cdot10^{-8}$ mol U per g of solution have been established by mass metrology. The methodology used in the preparation and certification was similar to that of comparable uranium reference materials made in the past [1,2]. The certified amount ratios were established by a TRITON TIMS using Faraday collectors and a secondary electron multiplier in combination with an RPQ energy filter for improved abundance sensitivity. Verification of the isotope amount content of IRMM-3660 was performed by IDMS using the TRITON TIMS.

The uncertainties contributing to the final uncertainties of the isotopic ratios are the weighing errors, the measured impurities in the starting material, the stoichiometry of the oxide and the isotopic abundances of the primary base materials. The method for the preparation is described and the certification procedure is reported.

The Isotopic Reference Materials IRMM-3660, IRMM-3660a and IRMM-3660b 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 [3]. Because of the extremely low abundance of ^{236}U in nature it can be very easily applied as a spike in uranium analysis by isotope dilution mass spectrometry.

In the frame of the IRMM programme to supply Isotopic Reference Materials a ^{236}U spike isotopic reference materials traceable to the SI has been prepared with the highest metrological quality for certified isotopic ratios and contents.

In this report the preparation and certification of a ^{236}U Spike Isotopic Reference Material produced by gravimetrical weighing of highly enriched 99.97% ^{236}U (IRMM-3660) is described.

3. Design

The primary solution IRMM-3660 is designed by gravimetrical weighing of a starting material 99.97% ^{236}U (BC02676). A further 10-fold dilution of the primary mixture was made to prepare IRMM-3660a; a 100-fold dilution of the primary mixture was made to prepare IRMM-3660b. The primary ^{236}U solution prepared and successfully used in the preparation of IRMM-075 series and also for the preparation of the IRMM-3636, a 233/236U Double Spike, was available as starting material.

[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.

4. Certification

The certification of the isotope ratios of the highly enriched ^{236}U starting material (BC02676) was accomplished by isotopic measurements using a Thermo-Electron Triton TIMS at IRMM. Isotopic measurements were performed using the modified total evaporation technique [4,5,6]. By using the principle of total evaporation the measurement is continued until the sample is exhausted in order to minimize mass fractionation effects.

Mass fractionation correction for all measured ratios of the starting materials was done using the close to natural uranium isotope reference material IRMM-184, with a certified $n(^{235}U)/n(^{238}U)$ ratio of 0.0072623(22). In order to avoid memory effects from the first plate of the ion source, the measurements of IRMM-184 were always performed after the measurements of the ^{236}U-enriched samples were completed. The correction was found to be relatively small (<0.02%) and the contribution to the uncertainty budgets of the isotopic composition of the ^{236}U starting materials is quite small. Certified values for the isotope amount ratios, amount contents and uncertainties were calculated according to ISO/GUM recommendations [7] using the GUM Workbench [8].

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

Certified values for the isotope ratios and the uncertainty budget with the major components of uncertainty are listed in Table 1.

Table 1: Isotopic composition of IRMM-3660, IRMM-3660a, IRMM-3660b

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) $g \cdot mol^{-1}$

The certified values for isotope amount contents and uncertainty budgets with the major components of uncertainty are listed in Table 2 and Table 3.

Table 2: Certified values for isotope content of IRMM-3660, 3660a and 3660b

Quantity	Value	
IRMM-3660		
U amount content($C_{U\text{-}3660}$):	4.23651(43)	µmol U/g
U mass content ($\gamma_{U\text{-}3660}$):	1.00001(10)	mg U/g
^{236}U isotope amount content ($C_{236U\text{-}3660}$):	4.23538(43)	µmol ^{236}U/g
^{236}U mass amount content ($\gamma_{236U\text{-}3660}$):	0.99974(10)	mg^{236}U /g
IRMM-3660a		
U amount content($C_{U\text{-}3660a}$):	0.422498(44)	µmol U/g
U mass content ($\gamma_{U\text{-}3660a}$):	99.729(10)	µg U/g
^{236}U isotope amount content ($C_{236U\text{-}3660a}$):	0.422385(44)	µmol ^{236}U/g
^{236}U mass amount content ($\gamma_{236U\text{-}3660a}$):	99.702(10)	µg^{236}U /g
IRMM-3660b		
U amount content($C_{U\text{-}3660b}$):	42.655(11)	nmol U/g
U mass content ($\gamma_{U\text{-}3660b}$):	10.0685(25)	µg U/g
^{236}U isotope amount content ($C_{236U\text{-}3660b}$):	42.643(11)	nmol ^{236}U/g
^{236}U mass amount content ($\gamma_{236U\text{-}3660b}$):	10.0658(25))	µg^{236}U /g

Table 3: Uncertainty budget for certified uranium amount contents

Quantity	Description	Value		%
IRMM-3660				
$f_{236oxide}$	stoichiometry base material	8.0		3.0
m_{U236O}	mass base material	158.970(3)	mg	4.6
$m_{sol3660}$	mass solution 3660	134.758(3)	g	6.4
evaporation correction	sampling from IRMM-3660	-		86.0
IRMM-3660a				
$f_{236oxide}$	stoichiometry base material	8.0		2.9
m_{U236O}	mass base material	158.970(3)	mg	4.5
$m_{sol3660}$	mass solution 3660	134.758(3)	g	6.2
evaporation correction	sampling from IRMM-3660	-		84.3
$m_{3660\text{-}for\text{-}3660a}$	mass IRMM-3660 for dilution	20.0322(2)	g	1.3
$m_{3660a\text{-}total}$	total IRMM-3660a	200.8687(16)	g	0.8

Quantity	Description	Value		%
IRMM-3660b				
$f_{236oxide}$	stoichiometry base material	8.0		0.5
m_{U236O}	mass base material	158.970(3)	mg	0.8
$m_{sol3660}$	mass solution 3660	134.758(3)	g	1.0
evaporation correction	sampling from IRMM-3660	-		14.3
$m_{3660\text{-}for\text{-}3660b}$	mass IRMM-3660 for dilution	1.0080(2)	g	83.2
$m_{3660a\text{-}total}$	total IRMM-3660b	100.1152(9)	g	0.2

For this material no verification measurements were envisaged in view of the use in the preparation of synthetic mixtures such as the series IRMM-075, a set of mixtures of natural uranium and ^{236}U, and in the double spike IRMM-3636. Extensive verification measurements have been performed on those materials which have shown intrinsically the correctness of the certified values of IRMM-3660 and therefore its dilutions.

5. Ampouling

Ampouling 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-3660), 100 µg (IRMM-3660a) and 10 µg (IRMM-3660b) 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 ampouling.

The flask containing the 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 4 below.

The same procedure was applied for the ampouling of both IRMM-3660a and IRMM-3660b.

From earlier experiments and testing carried out during similar operations [9], 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-3660, 3660a or 3660b solutions.

Table 4: Sequence of ampoule filling for IRMM-3660, 3660a and 3660b

	Date	Number of ampoules sealed
3660	10-06-2008	33
3660a	18-03-2008	196
3660b	17-03-2008	95

6. Conclusions

The methodology and techniques used in the preparation of synthetic mixtures IRMM-074, IRMM-075 and IRMM-3636 have again been applied successfully for the preparation of IRMM-3660 and of its dilutions IRMM-3660a and b.

The materials have been prepared and certified values of the isotope amount ratios and contents have been calculated based on the weights of oxides and solutions.

The isotopic reference material IRMM-3660 and IRMM-3660a and b are commercially available from IRMM.

7. References

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[7] International Organisation for Standardisation, Guide to the Expression of Uncertainty in Measurements, ©ISO, ISBN 92-67-10188-9, Geneva, Switserland 1993

[8] GUM Workbench, Metrodata GmbH, www.metrodata.de

[9] A. Held, P. Taylor, A. Verbruggen, R. Wellum, ”IRMM-073 Certification Report”, Internal report GE/R/IM/22/02

Figure 1: Certificate IRMM-3660

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Isotope Measurements (Geel)

CERTIFICATE
SPIKE ISOTOPIC REFERENCE MATERIAL IRMM-3660

$4.235\ 38(43) \cdot 10^{-6}$ 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:

$n(^{233}U)/n(^{236}U)$:	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)

This corresponds to an isotopic composition with the following abundances:

	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 in this sample is 236.045 971 7(43) g·mol^{-1}

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

$4.236\ 51(43) \cdot 10^{-6}$	mol (U) · g^{-1} (solution)
$1.000\ 01(10) \cdot 10^{-3}$	g (U) · g^{-1} (solution)
$0.999\ 74(10) \cdot 10^{-3}$	g (^{236}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 8 May 2006.

5. The Isotopic Reference Material IRMM-3660 comes in a flame-sealed quartz ampoule containing about 4.2 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 23406 EN[3]

Chemical purification of the $^{236}U_3O_8$ starting material 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-3660a 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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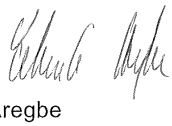
[1] International Organisation for Standardisation, Guide to the expression of Uncertainty in Measurement, ©ISO, ISBN 92-67-10188-9, Geneva, Switzerland, 1993
[2] G. Audi and A.H. Wapstra, The 2003 atomic mass evaluation, Nucl Phys A729(2003) 337-676.
[3] A. Verbruggen, A. Alonso, R. Eykens, F. Kehoe, H. Kühn, S. Richter, Y. Aregbe, Preparation and certification of IRMM-3660, 3660a and 3660b, Report EUR 23406 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
July 2008	Head Isotope Measurements Unit	IRMM Safeguards Coordinator

Figure 2: Certificate IRMM-3660a

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CERTIFICATE
SPIKE ISOTOPIC REFERENCE MATERIAL IRMM-3660a

4.223 85(44) · 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:

$n(^{233}U)/n(^{236}U)$:	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)

This corresponds to an isotopic composition with the following abundances:

	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 in this sample is 236.045 971 7(43) $g \cdot mol^{-1}$

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

4.224 98(44) · 10^{-7}	mol (U) · g^{-1} (solution)
99.729(10) · 10^{-6}	g (U) · g^{-1} (solution)
99.702(10) · 10^{-6}	g (^{236}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 8 May 2006.

5. The Isotopic Reference Material IRMM-3660a 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 23406 EN [3]

Chemical purification of the $^{236}U_3O_8$ starting material 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-3660a 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.
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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.

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July 2008	Head Isotope Measurements Unit	IRMM Safeguards Coordinator

Figure 3: Certificate IRMM-3660b

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CERTIFICATE
SPIKE ISOTOPIC REFERENCE MATERIAL IRMM-3660b

4.264 3(11) · 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 amounts of other uranium isotopes present are related to the ^{236}U content through the following certified amount ratios:

$n(^{233}U)/n(^{236}U)$:	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)

This corresponds to an isotopic composition with the following abundances:

	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 in this sample is 236.045 971 7(43) $g \cdot mol^{-1}$

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

4.265 5(11) · 10^{-8}	mol (U) · g^{-1} (solution)
10.068 5(25) · 10^{-6}	g (U) · g^{-1} (solution)
10.065 8(25) · 10^{-6}	g (^{236}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 8 May 2006.

5. The Isotopic Reference Material IRMM-3660b comes in a flame-sealed quartz ampoule containing about 42 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) $g \cdot mol^{-1}$
^{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 23406 EN [3]

Chemical purification of the $^{236}U_3O_8$ starting material 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-3660a 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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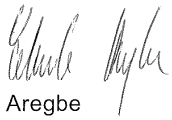
[1] International Organisation for Standardisation, Guide to the expression of Uncertainty in Measurement, ©ISO, ISBN 92-67-10188-9, Geneva, Switzerland, 1993
[2] G. Audi and A.H. Wapstra, The 2003 atomic mass evaluation, Nucl Phys A729(2003) 337-676.
[3] A. Verbruggen, A. Alonso, R. Eykens, F. Kehoe, H. Kühn, S. Richter, Y. Aregbe, Preparation and certification of IRMM-3660, 3660a and 3660b, Report EUR 23406 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.



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P Taylor
Head
Isotope Measurements Unit

Y Aregbe
IRMM Safeguards Coordinator

European Commission

EUR 23406 EN – Joint Research Centre – Institute for Reference Materials and Measurements

Title: Preparation and Certification Report IRMM-3660, IRMM-3660a and IRMM-3660b

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

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Abstract

Isotope reference materials IRMM-3660 was prepared by dissolution of highly enriched 99.97% ^{236}U in nitric acid. IRMM-3660a and IRMM-3660b were prepared from IRMM-3660 by gravimetrical dilution.

The certified isotope content values for IRMM-3660 of $4.23651(43)\cdot10^{-6}$ mol U per g of solution, IRMM-3660a of $4.22498(44)\cdot10^{-7}$ mol U per g of solution and for IRMM-3660b of $4.2655(11)\cdot10^{-8}$ mol U per g of solution have been established by mass metrology. The methodology used in the preparation and certification was similar to that of comparable uranium reference materials made in the past. The certified amount ratios were established by a TRITON TIMS using Faraday collectors and a secondary electron multiplier in combination with an RPQ energy filter for improved abundance sensitivity. Verification of the isotope amount content of IRMM-3660 was performed by IDMS using the TRITON TIMS.

The uncertainties contributing to the final uncertainties of the isotopic ratios are the weighing errors, the measured impurities in the starting material, the stoichiometry of the oxide and the isotopic abundances of the primary base materials. The method for the preparation is described and the certification procedure is reported.

The Isotopic Reference Materials IRMM-3660, IRMM-3660a and IRMM-3660b 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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