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Certification Report

REIMEP 18 Inter-Laboratory Comparison for the Measurement of Uranium Isotopic Ratios in Nitric Acid Solution

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Abstract

The REIMEP 18 (Regular European Inter-laboratory Measurement Evaluation Programme) campaign was started in October 2005. For this campaign 4 samples of depleted to low-enriched uranium were chosen. Certification measurements were carried out using a Varian MAT511 UF_6-gas source mass spectrometer (GSMS) for the $^{235}U/^{238}U$ ratio and using a Triton thermal ionisation mass spectrometer (TIMS) for the minor isotope ratios $^{234}U/^{238}U$ and $^{236}U/^{238}U$. Verification measurements of ampouled samples were performed successfully and showed good agreement with the certified ratios.
REIMEP 18 has become one of the largest nuclear measurement campaigns organized by IRMM so far. Samples were shipped in March 2006 to ca. 80 registered participants. The participating laboratories are expected to submit their results until May 31st, 2006. This report describes the planning of the REIMEP 18 campaign, including the selection of campaign samples and the certification measurements.

Introduction

In October 2005 the REIMEP (Regular European Inter-laboratory Measurement Evaluation Programme) campaign 18 for the measurement of isotopic ratios of uranium in nitric acid solution was started. This campaign was organized in order to respond to the needs for external quality control expressed by customers from the nuclear safeguards and the scientific area.
For the REIMEP 18 campaign 4 samples of depleted to low-enriched uranium were selected from the IRMM stock. Each of the 4 samples contains 2.5mg uranium in 0.5mL of 0.5M nitric acid solution. The sample amounts were chosen in order to achieve a total activity below 1000Bq for each set of 4 samples, which allows the sample sets to be considered and shipped as non-nuclear material. This is a significant advantage for a large number of participating laboratories, because extensive administrative work related to nuclear transport requirements could be avoided. As a consequence REIMEP 18 has become one of the largest nuclear measurement campaigns ever organized by IRMM. About 80 laboratories have registered to participate, about 60% coming from the nuclear safeguards and 40% from the scientific area, among those mainly from geology/geochemistry.

The lines of the previous REIMEP campaigns were followed:

1. IRMM supplied the samples 'blind', i.e. without certified isotopic abundance values.
2. A participating fee of € 200 is charged per laboratory (dispatch costs are included) except for participants from the new EU member states, the Accession and Western Balkan countries (Albania, Bulgaria, Bosnia-Herzegovina, Cyprus, Croatia, Czech Republic, Estonia, Hungary, Latvia, Lithuania, FYR of Macedonia, Malta, Poland, Rumania, Serbia- Montenegro, Slovakia, Slovenia and Turkey.
3. Each laboratory is asked to measure the $^{234}U/^{238}U$, $^{235}U/^{238}U$ and $^{236}U/^{238}U$ isotope ratios, the ^{234}U, ^{235}U, ^{236}U and ^{238}U abundances and mass fractions. The results are entered by the participants into the REIMEP 18 database via the IRMM website.
4. Each laboratory has been supplied with an identification number and all results will be published using this number; IRMM will not disclose laboratories' identities without direct permission from the laboratories concerned.
5. On receipt of the measured values IRMM will send the certification report including the certified values (see appendix) to the laboratory.
6. IRMM will publish a participants' report showing in a graphical form the results relative to the certified values.

Certification Measurements for REIMEP 18

The original uranium samples chosen for the REIMEP 18 campaign were in UF_6 form. They were measured and certified for the major ratio $^{235}U/^{238}U$ using a Varian MAT511 UF_6-gas source mass spectrometer (GSMS), which is calibrated using synthetic isotope mixtures. Due to the very high reproducibility of the GSMS instrument typically combined uncertainties of about 0.02%-0.06% (coverage factor k=2) are obtained, depending on the ratio. For certification measurements at IRMM usually an "augmented" uncertainty of at least 0.05% (coverage factor k=2) is provided, because there is

not yet a need for $^{235}U/^{238}U$ ratio measurements with uncertainties smaller than 0.05%, this may well change in the future.
The samples in UF_6 form were hydrolyzed and calcined in order to obtain the uranium in oxide form (mainly U_3O_8). The oxides were dissolved in nitric acid to obtain the "mother solutions" for REIMEP 18 A-D. In order to verify the certified major ratios $^{235}U/^{238}U$ for REIMEP 18 A-D, TIMS measurements were performed using the "Modified Total Evaporation" (MTE) technique as described in [1]. The results are presented in Figure 1 and show an good agreement with the certified values obtained using GSMS.

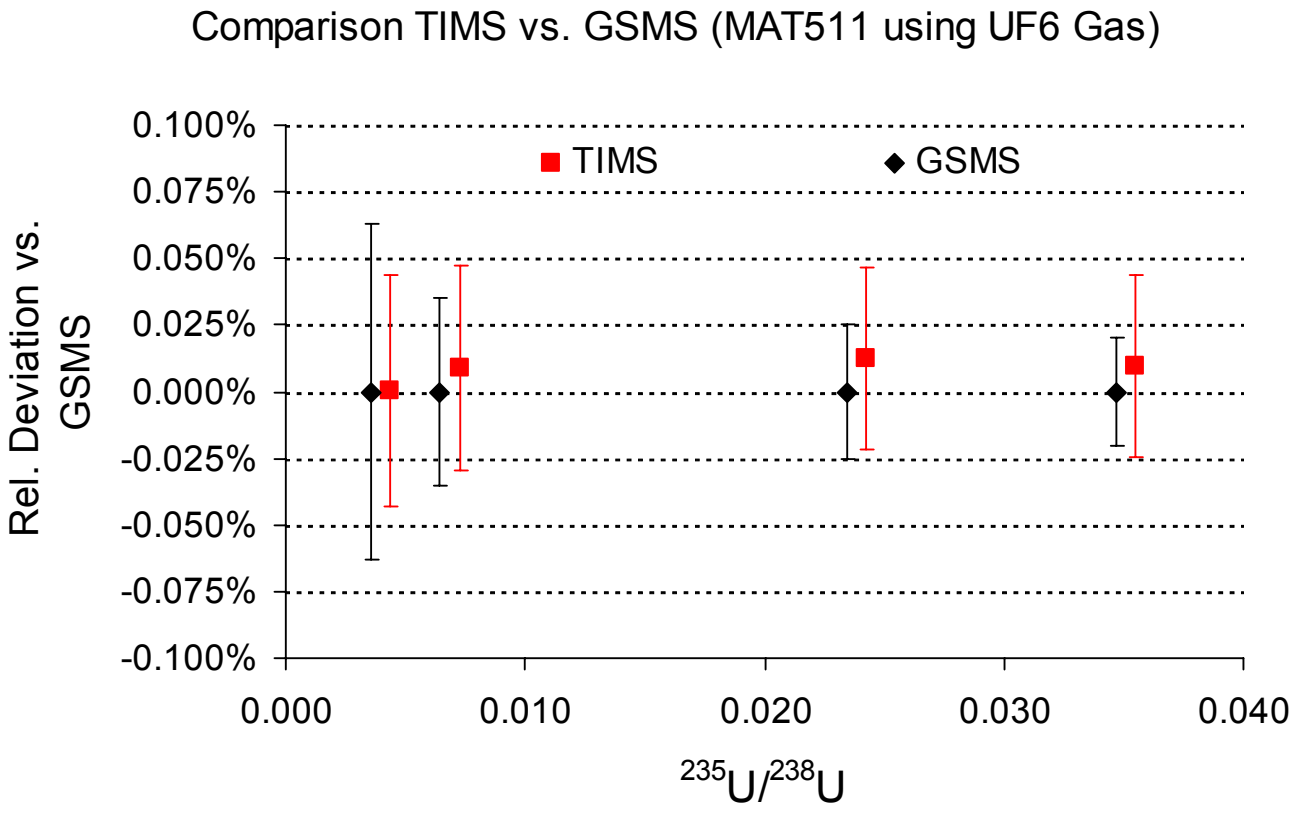

Fig.1: Comparison of $^{235}U/^{238}U$, between TIMS verification measurements and certified data obtained by GSMS.

The minor isotope ratios, $^{234}U/^{238}U$ and $^{236}U/^{238}U$ were then measured and certified using a Triton thermal ionization mass spectrometer (TIMS). The so-called HI-method (HI="high intensity") is described in detail in references [1-4], and will only be recapitulated briefly here.

To measure the minor isotope ratios $^{234}U/^{238}U$ and $^{236}U/^{238}U$ for REIMEP 18 the double filament technique was applied as for most elements with high ionization potential. For each sample three 1μl drops of a 1 molar nitric solution containing 5μg uranium were dried down on a zone-refined rhenium filament, which had been degassed for 30 minutes at 1800^0C to remove impurities. All prepared sample filaments together with the corresponding ionization filaments were mounted on a 21 position sample turret.
Because of the extended dynamic range and improved signal-to-noise ratio of the Faraday multi-collector of the new Triton TIMS all $^{234}U/^{238}U$ ratios of the REIMEP 18 materials could be measured in Faraday mode. A measurement of the intensity at mass 234 using an SEM (secondary electron multiplier) would introduce additional uncertainty contributions, arising from the linearity correction of the SEM [5] and the inter-calibration between the SEM and the Faraday multi-collector. In order to further decrease the uncertainties for the $^{234}U/^{238}U$ ratios measured using Faraday cups by a factor of about 2, a special amplifier with a $10^{12}\Omega$ resistor was utilized for the Faraday cup for ^{234}U, in spite of the standard $10^{11}\Omega$ resistor as e.g. used for the ^{235}U and ^{238}U Faraday cups.
The ion beam intensity of ^{236}U is only sufficient for Faraday cup detection for $^{236}U/^{238}U$ ratios $>10^{-5}$, in the case of REIMEP 18 this only applies to the samples REIMEP 18 B and C. For these samples also the

Faraday cup for ^{236}U was equipped with a special amplifier with a $10^{12}\Omega$ resistor in order to improve the signal-to-noise ratio.
For the 2 other materials, REIMEP 18 A and D, the ^{236}U had to be measured using the SEM in ion-counting mode. The inter-calibration between the SEM and the Faraday multi-collector was done using the ^{234}U beam switched between a Faraday cup and the SEM. Because the ^{234}U intensity on the Faraday cup is quite low, the correction of the ^{234}U intensity for the tailing contributions from the much higher ion beams of ^{235}U and ^{238}U is significant for an accurate SEM/Faraday inter-calibration.
Within the SEM ion counting detector there are tailing contributions for both minor isotopes ^{234}U and ^{236}U as well, originated from the much higher ion beams of ^{235}U and ^{238}U, but all tailing effects observed in the SEM are generally reduced by a factor of about 100 using an energy filter in front of the SEM detector. For the ^{234}U detection during the SEM/Faraday inter-calibration (count rate ca. 100.000 counts per second) the tailing effect contribution is negligible, but for measuring $^{236}U/^{238}U$ ratios in the order of 10^{-8} - 10^{-6} using the SEM ion counter there is a significant tailing contribution in the order of about 10^{-11} -10^{-10}, which has to be corrected for.

The ^{234}U intensity on the Faraday cup and the ^{236}U on the SEM are corrected regularly for the tailing effects during the measurement. The entire measurement consists of 8-10 blocks of 5 mass cycles each. For every mass cycle the tailing effects are measured at masses 233.7 and 234.4 to provide an average tailing contribution at ^{234}U (mass = ca. 234.05 u), and at masses 235.7 and 236.4 to provide an average tailing contribution at ^{236}U (mass = ca. 236.05 u). Also the SEM/Faraday inter-calibration is performed for every mass cycle throughout the entire measurement time, which provides more reliable results than in a procedure in which the inter-calibration is only done once at the beginning prior to the measurement. The mass cycle is arranged as follows:

Step	Cup L2	Cup L1	Cup C, **SEM**	Cup H1	Cup H2	Integration time (s)	Idle time (s)
1	234	235	236		238	64	15
2			234	235		8	2
3	233.7	234.7	235.7		237.7	32	15
4	234.4	235.4	236.4		238.4	32	2

Step 2 is (only) introduced to measure ^{234}U in the SEM ion counter in order to calibrate the ion counter against the Faraday multi-collector. The relatively long idle times (15s) are necessary because of the slow response of the special amplifier with a $10^{12}\Omega$ resistor; similar waiting times have to observed prior to each measurement of the electronic amplifier baseline or gain.
The fractionation correction for the $^{234}U/^{238}U$ and $^{236}U/^{238}U$ ratios is also performed for each mass cycle throughout the entire measurement time, calculated internally using the $^{235}U/^{238}U$ value measured by UF_6-gas source mass spectrometry. Combined uncertainties of the measurement results for REIMEP 18 A-D were calculated according to the “Guide to the Expression of Uncertainty in Measurements” [7]. They include all sources of uncertainty originating from the corrections for fractionation, linearity of the SEM and inter-calibration of the SEM versus the Faraday collector

In order to control the accuracy of measured isotope ratios over a wide dynamic range suitable isotope reference materials have to be used. For example the IRMM-072 series of reference materials [8] (and its replacement IRMM-074, [9]), provides certified isotope ratios ranging from 1 down to 10^{-6} for the $^{233}U/^{238}U$ ratio and a $^{235}U/^{238}U$ ratio of 1. Therefore IRMM-072 and IRMM-074 were used to verify the linearity for the Faraday multi-collector of the Triton TIMS. This is needed as a proof for the reliability of the $^{234}U/^{238}U$ measurements using the entire dynamic range of the Faraday multi-collector. The IRMM-074 series was also used to establish the parameters for a linearity correction of the SEM ion counter, as described in [5].
Furthermore a set of three synthetic calibration mixtures with calculated $^{236}U/^{238}U$ ratios of 10^{-6}, 10^{-7}, and 10^{-8} was prepared to serve for an additional validation specifically for measurements of ^{236}U in ion counting mode [6]. These calibration mixtures were composed of one natural uranium starting material (^{236}U below detection limit) and one non-natural starting material for which the ^{236}U isotope intensity could be measured using a Faraday collector alone.
Samples of these 3 synthetic mixtures with certified $^{236}U/^{238}U$ ratios of 10^{-6}, 10^{-7}, and 10^{-8} were measured on the same sample turrets along with the samples REIMEP 18 A and D. This provided an additional

quality control for the technique to measure $^{236}U/^{238}U$ ratios below 10^{-5} using the SEM ion counter to detect ^{236}U.
The certification results for the samples REIMEP 18 A-D are presented by their certificates in the appendix. After the certification was completed, 100 sample sets for REIMEP 18 A-D were prepared and shipped to all registered participants. On one REIMEP 18 A-D sample set additional verification measurements were performed at IRMM using TIMS only and showed good agreement with the certified values.

Conclusions

The sample preparation, certification and verification measurements for REIMEP 18 were completed successfully. Mass spectrometric certification measurements for the REIMEP 18 campaign were performed at IRMM with advanced technology for uranium isotope ratio measurements, leading to state of the art precision and accuracy in isotopic measurements.
Samples have been shipped to ca. 80 laboratories worldwide, including areas such as nuclear safeguards, nuclear forensics, environmental and geological research, which are using various mass spectrometric techniques such as TIMS, Quadrupole-ICP-MS, HR-ICP-MS, MC-ICP-MS, AMS, RIMS, α-spectrometry, etc. Therefore the REIMEP 18 inter-laboratory comparison is expected to provide a very meaningful and highly representative assessment of the current capabilities for uranium isotope measurements on a worldwide scale.

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Appendix:

Certificates for samples REIMEP 18 A-D

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CERTIFICATE of a reference measurement

IM/MeaC/40/05-REIMEP 18-A
1 December 2005

1. Applicant: R Wellum, IRMM

2. Sample Identification:
   - REIMEP 18-A, LOT 2415
   - Chemical forms: UF_6, Uranium Nitrate
   - IM sample registration number: IMN-10165

3. Measurands:
   - Isotopic composition

isotope amount ratio(s)	
$n(^{234}U)/n(^{238}U)$	0.000 056 582(41)
$n(^{235}U)/n(^{238}U)$	0.007 254 2(36)
$n(^{236}U)/n(^{238}U)$	0.000 000 030 579(83)

amount fraction (·100)		mass fraction (·100)	
$n(^{234}U)/n(U)$	0.005 617 2(40)	$m(^{234}U)/m(U)$	0.005 523 1(40)
$n(^{235}U)/n(U)$	0.720 16(36)	$m(^{235}U)/m(U)$	0.711 12(35)
$n(^{236}U)/n(U)$	0.000 003 035 7(82)	$m(^{236}U)/m(U)$	0.000 003 010 4(82)
$n(^{238}U)/n(U)$	99.274 22(36)	$m(^{238}U)/m(U)$	99.283 35(36)

molar mass: 238.028 904(12) $g \cdot mol^{-1}$

4. Date of receipt of sample : 10 November 2005
   Date of completion of measurement : 18 November 2005

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5. Uncertainty:
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.
The primary certified values are the isotope amount ratios; other values are derived from them. Reproducing the derived values may result in differences due to rounding errors.

6. The traceability to SI is established through gravimetrically prepared standards from IRMM.

7. Analytical measurement procedure

- Mass spectrometric measurements were performed by W De Bolle for the $[n(^{235}U)/n(^{238}U)]$ isotope ratio using the MAT511 mass spectrometer on UF_6 samples prepared by W De Bolle. TIMS measurements on $[n(^{234}U)/n(^{238}U)]$ and $[n(^{236}U)/n(^{238}U)]$ were performed by S Richter using the TRITON mass spectrometer, sample solutions were prepared for TIMS analysis by A Alonso. A Verbruggen was responsible for the preparation and issuance of the certificate.
- The atomic masses, used in the calculations, are from G. Audi and A.H. Wapstra, The 1993 atomic mass evaluation , Nucl Phys A565 (1993) 1-65.
- Reference number of the measurement data: measurement number T5B18, logged in S:\Im UNIT\Secure Data\Archive MS Measurements data files\TRITON\data.

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CERTIFICATE of a reference measurement

IM/MeaC/41/05-REIMEP 18-B
1 December 2005

1. Applicant: R Wellum, IRMM

2. Sample Identification:
   - REIMEP 18-B, LOT 2419
   - Chemical forms: UF_6, Uranium Nitrate
   - IM sample registration number: IMN-10167

3. Measurands:
   - Isotopic composition

isotope amount ratio(s)	
$n(^{234}U)/n(^{238}U)$	0.000 332 71(22)
$n(^{235}U)/n(^{238}U)$	0.035 470(18)
$n(^{236}U)/n(^{238}U)$	0.000 388 28(13)

amount fraction (·100)		mass fraction (·100)	
$n(^{234}U)/n(U)$	0.032 109(21)	$m(^{234}U)/m(U)$	0.031 582(21)
$n(^{235}U)/n(U)$	3.423 1(17)	$m(^{235}U)/m(U)$	3.381 4(16)
$n(^{236}U)/n(U)$	0.037 472(12)	$m(^{236}U)/m(U)$	0.037 173(12)
$n(^{238}U)/n(U)$	96.507 3(17)	$m(^{238}U)/m(U)$	96.549 9(17)

molar mass: 237.945 816(51) $g \cdot mol^{-1}$

4. Date of receipt of sample : 10 November 2005
   Date of completion of measurement : 25 November 2005

5. Uncertainty:
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.
The primary certified values are the isotope amount ratios; other values are derived from them. Reproducing the derived values may result in differences due to rounding errors.

6. The traceability to SI is established through gravimetrically prepared standards from IRMM.

7. Analytical measurement procedure

- Mass spectrometric measurements were performed by W De Bolle and J Truyens for the $[n(^{235}U)/n(^{238}U)]$ isotope ratio using the MAT511 mass spectrometer on UF_6 samples prepared by W De Bolle. TIMS measurements on $[n(^{234}U)/n(^{238}U)]$ and $[n(^{236}U)/n(^{238}U)]$ were performed by S Richter using the TRITON mass spectrometer, sample solutions were prepared for TIMS analysis by A Alonso. A Verbruggen was responsible for the preparation and issuance of the certificate.
- The atomic masses, used in the calculations, are from G. Audi and A.H. Wapstra, The 1993 atomic mass evaluation , Nucl Phys A565 (1993) 1-65.
- Reference number of the measurement data: measurement number T5B22, logged in S:\Im UNIT\Secure Data\Archive MS Measurements data files\TRITON\data.

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CERTIFICATE of a reference measurement

IM/MeaC/42/05-REIMEP 18-C
1 December 2005

1. Applicant: R Wellum, IRMM

2. Sample Identification:
   - REIMEP 18-C, LOT 2426
   - Chemical forms: UF_6, Uranium Nitrate
   - IM sample registration number: IMN-10168

3. Measurands:
   - Isotopic composition

isotope amount ratio(s)	
$n(^{234}U)/n(^{238}U)$	0.000 079 510(68)
$n(^{235}U)/n(^{238}U)$	0.004 379 4(27)
$n(^{236}U)/n(^{238}U)$	0.001 03370(44)

amount fraction (·100)		mass fraction (·100)	
$n(^{234}U)/n(U)$	0.007 907 6(67)	$m(^{234}U)/m(U)$	0.007 774 9(66)
$n(^{235}U)/n(U)$	0.435 55(27)	$m(^{235}U)/m(U)$	0.430 07(26)
$n(^{236}U)/n(U)$	0.102 806(44)	$m(^{236}U)/m(U)$	0.101 946(43)
$n(^{238}U)/n(U)$	99.453 74(32)	$m(^{238}U)/m(U)$	99.460 20(31)

molar mass: 238.035 309(10) $g \cdot mol^{-1}$

4. Date of receipt of sample : 10 November 2005
   Date of completion of measurement : 25 November 2005

5. Uncertainty:
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.
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7. Analytical measurement procedure

- Mass spectrometric measurements were performed by W De Bolle for the $[n(^{235}U)/n(^{238}U)]$ isotope ratio using the MAT511 mass spectrometer on UF_6 samples prepared by W De Bolle. TIMS measurements on $[n(^{234}U)/n(^{238}U)]$ and $[n(^{236}U)/n(^{238}U)]$ were performed by S Richter using the TRITON mass spectrometer, sample solutions were prepared for TIMS analysis by A Alonso. A Verbruggen was responsible for the preparation and issuance of the certificate.
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CERTIFICATE of a reference measurement

IM/MeaC/43/05-REIMEP 18-D
1 December 2005

1. Applicant: R Wellum, IRMM

2. Sample Identification:
   - REIMEP 18-D, LOT 2417
   - Chemical forms: UF_6, Uranium Nitrate
   - IM sample registration number: IMN-10166

3. Measurands:
   - Isotopic composition

isotope amount ratio(s)	
$n(^{234}U)/n(^{238}U)$	0.000 209 36(14)
$n(^{235}U)/n(^{238}U)$	0.024 233(12)
$n(^{236}U)/n(^{238}U)$	0.000 000 110 54(29)

amount fraction (·100)		mass fraction (·100)	
$n(^{234}U)/n(U)$	0.020 436(14)	$m(^{234}U)/m(U)$	0.020 098(13)
$n(^{235}U)/n(U)$	2.365 5(12)	$m(^{235}U)/m(U)$	2.336 3(11)
$n(^{236}U)/n(U)$	0.000 010 790(28)	$m(^{236}U)/m(U)$	0.000 010 702(28)
$n(^{238}U)/n(U)$	97.614 1(12)	$m(^{238}U)/m(U)$	97.643 6(12)

molar mass: 237.978 837(36) $g \cdot mol^{-1}$

4. Date of receipt of sample : 10 November 2005
   Date of completion of measurement : 18 November 2005

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REIMEP 18 has become one of the largest nuclear measurement campaigns organized by IRMM so far. Samples were shipped in March 2006 to ca. 80 registered participants. The participating laboratories are expected to submit their results until May 31st, 2006. This report describes the planning of the REIMEP 18 campaign, including the selection of campaign samples and the certification measurements.

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