JRC Scientific and Technical Reports

Preparation and Certification of IRMM-1027j, Large-Sized Dried (LSD) spike

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S. Richter, Y. Aregbe

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JRC
EUROPEAN COMMISSION

irmm
Institute for Reference Materials and Measurements

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

European Commission
Joint Research Centre
Institute for Reference Materials and Measurements

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Contents

Summary

A new set of Large Size Dried (LSD) Spikes for the determination of uranium and plutonium by isotope dilution mass spectrometry in solutions of spent fuel from reprocessing plants has been prepared and certified for uranium and plutonium isotopic contents. The methodology followed was similar to that of previous batches. The solution, made by dissolution of the starting materials in nitric acid, was dispensed directly into individual penicillin vials.

This new batch of large size dried spikes contains ca. 50 mg of uranium (^{235}U abundance = 19.7%) and ca. 1.8 mg of plutonium (^{239}Pu abundance = 97.8%) in each individual vial, covered with a light layer of organic material (cellulose acetate butyrate) as stabilizer.

The U and Pu amount content was certified based on values from mass metrology. Verification of the amount contents of the spike was done by IDMS at IRMM. The values measured for the batch solution and of the dried covered spikes agreed well with those calculated from the weights of starting materials dissolved and the weights of the final solution.

Introduction

The series IRMM-1027j Large Size Dried (LSD) Spikes is being prepared to fulfil the existing requirement for reliable and traceable spikes in fissile material control of dissolved nuclear fuel. The amount content of the spikes is such that no dilution of a typical sample of dissolved fuel is needed before measurement by Isotope Dilution Mass Spectrometry (IDMS) using a single LSD spike. Because each spike is certified for amounts of plutonium and uranium in the vial, the only quantitative step needed at the reprocessing plant laboratory is to weigh as accurately as possible an aliquot of the dissolved fuel solution onto the spike and ensure complete mixing of spike and sample.

The plutonium component is highly enriched in ^{239}Pu and is used to measure the Pu content in the fuel. Approximately 1.8 mg Pu is contained in each LSD spike. The uranium component is a mixture of two uranium source materials, natural uranium and a highly enriched uranium component. These materials are mixed to arrive at a final enrichment of just under 20% in ^{235}U, which means for accountability purposes the uranium is classified as ‘low enriched’.

High purity metals are chosen as starting materials. For this campaign it was decided to use CETAMA MP2 metal as in most previous batches. This allows the isotopic contents of the LSD spike to be certified from the certificates of the metals (chemical purity and isotopic content), the weights of the metals and the solution. As a result the values of the uranium and plutonium isotopic contents of the final certified spike solution have low uncertainties which are directly traceability to the SI via the weights of the starting materials.

A single large volume of batch solution is made up and dispensed into a number of penicillin vials. The solution in each vial is dried down and then covered with a light organic coating dried onto the spike material. The coating (cellulose acetate butyrate, CAB) that was used for previous batches has excellent physical and chemical properties for this purpose. It provides a fixed layer to hold the dried spike material on the base of the vial, dissolves quickly in warm nitric acid and has no significant effect on the subsequent IDMS measurements.

Following the experience of previous series, in particular IRMM-1027e, 1027f, 1027g, 1027h and 1027i, the isotopic contents of the batch solution and of a set of individual spike vials after drying are measured by isotope dilution to verify the values from the mass-metrology of the starting metals dissolved and the weight of the final solution.

Dissolution of standard materials

Pu Metal Cetama MP2

The metal standard is delivered in a flame-sealed vial with a certified mass of Pu metal. Four vials were required for the preparation of this LSD spike. Each vial was cut open, the Pu removed with tweezers, weighed and placed in the 3 L borosilicate flask (see next paragraph). The total amount of Pu, calculated to obtain a solution of ca. 0.7 mg plutonium per gram solution when dissolved in 3 kg nitric acid, was weighed at IRMM. The weight of metal agreed well with the certified mass of the MP2 metal

Uranium metals EC NRM 101, CRM-116

Approximately 47.4 g EC NRM 101 (natural uranium) metal was etched with 1 M HNO_3 as recommended on the certificate to remove surface oxides, rinsed with deionised water then acetone and finally dried. The metal was accurately weighed and added to the flask containing the Pu solution. The same was done with 12.2 g NBL CRM-116 enriched uranium. The masses of the uranium were calculated so as to yield a solution of ca. 20 mg uranium per gram solution with an enrichment of ca. 19.8% in ^{235}U.

Making up the batch solution

The dissolution was carried out entirely in a 3 L long-necked borosilicate flask that had been cleaned in the IRMM MCL (Medium-Clean Chemistry Laboratory). All weighings were carried out as accurately as possible, with reference to a set of calibrated weights traceable to the kilogram at BIPM, Sèvres. The necessary corrections for buoyancy effects, taking into account the ambient pressure, temperature, humidity and the density of the material were made.

The weighed Pu metal was transferred into the flask. Concentrated nitric acid and a few drops of conc. HF were added and the flask was warmed to about 90° C to dissolve the Pu. The dissolution was controlled visually and took several weeks to be complete. After cooling the solution was kept under controlled conditions to ensure complete Pu dissolution before the uranium was added. The uranium dissolved quickly and completely within a few days.

The complete dissolution of the metals and the solution homogeneity was ensured by allowing the solution to stand for at least 8 weeks after the starting materials had been adjudged to be completely dissolved.

After making up the solution to the prescribed weight of 3.1 kg, the solution was left for another 4 weeks to homogenise with occasional swirling by hand.

Measurement of isotopic abundances in the batch solution

A plastic syringe (50 mL) was filled from the batch solution and from this syringe 6 aliquots of 1 g were weighed into a set of glass vials. These were then spiked with 5 g each of IRMM-046b double spike (^{233}U+^{242}Pu) for isotope dilution mass spectrometry (IDMS). One extra vial containing ca. 1 g of solution was not spiked and was processed for measurement of isotopic ratios.

The chemical procedure prior to mass spectrometry as detailed in [1] was employed. A 1 M HNO_3 solution of uranium and of plutonium separated from the spiked and unspiked solutions were prepared for measurements of the isotopic ratios by TIMS.

The isotopic ratios of the uranium were measured on the Finnigan Triton and those of plutonium on the Finnigan MAT 262, following IRMM Quality Management procedures PR-077 for uranium and PR-075 for plutonium. The mass-spectrometers were calibrated for mass-fractionation by measuring IRMM-184 uranium isotopic reference material and IRMM-290 plutonium isotopic reference material during the procedure.

The measured ratios compared to the calculated values from the certificates are listed in Table 1 for uranium and Table 2 for plutonium. The certified ratios for uranium are taken from the Triton measurements and are compared to the ratios calculated from the mixing of the two metals and their certified isotopic abundances. The certified ratios for Pu are taken from the recertification of MP2 at IRMM (2007) as in the IRMM certificate in Annex 3.

Table 1: Isotopic amount ratios of uranium in the batch solution. Values from certificates and metrological weighing are compared with abundances calculated from measurement of isotopic ratios in a sample of the batch solution. Expanded Uncertainties are given in brackets (coverage factor *k*=2).

	$n(^{234}U)/n(^{238}U)$	$n(^{235}U)/n(^{238}U)$	$n(^{236}U)/n(^{238}U)$
Calculated value	0.002 624(11)	0.247 668(57)	0.001 080 0(33)
Measured/Certified value	0.002 623 8(12)	0.247 738(80)	0.001 082 03(70)

Table 2: Isotopic amount ratios of plutonium in the batch solution. Values from the certificate are compared with certified values calculated from measurement of isotopic ratios in a sample of the batch solution. Expanded Uncertainties are given in brackets (coverage factor *k*=2).

	$n(^{238}Pu)/n(^{239}Pu)$	$n(^{240}Pu)/n(^{239}Pu)$	$n(^{241}Pu)/n(^{239}Pu)$	$n(^{242}Pu)/n(^{239}Pu)$
Certified value	0.000 030 83(29)	0.022 432 4(51)	0.000 237 8(31)	0.000 075 70(78)
Measured value	0.000 031 12(14)	0.022 430 3(67)	0.000 238 96(75)	0.000 074 90(38)

Verification of U and Pu amounts in the batch solution

The series of 6 spiked solutions described above together with the vial containing the unspiked sample were heated to dryness, then chemically conditioned and the U and Pu fractions separated by the standard ion-exchange method (*Work Instructions: 042 'Spiking, isotopic exchange and preliminary separation for mixtures of uranium and plutonium'; 041 'Separation and purification of uranium for measurement of isotopic ratios by TIMS'; 035 'Separation of Pu for TIMS measurements of isotopic ratios for IDMS or for isotopic abundances'*).

For the plutonium, a magazine was loaded with one filament for each blend; the remaining positions were filled by IRMM-290 standards. The plutonium was measured on the MAT 262 (*Work Instruction: 115 'Pu isotopic measurements in total evaporation using the MAT 262'*). The uranium measurements were carried out on the Triton (*Working Instruction 149 'measurement of uranium isotopic ratios by the TIMS TRITON'*). One filament per blend was loaded; the remaining positions per magazine were loaded with the unspiked sample (6x) and IRMM-184 U isotopic reference material. The Triton measurements were carried out using the Modified Total Evaporation technique. The method is also described in detail in [2].

The results of these IDMS measurements are given in Table 3 and Table 4 and shown in Figs. 1 and 2 compared with the U and Pu amount contents as calculated from the masses of the dissolved metals and solutions, taking into account the certified chemical purities of the starting materials and making corrections for isotopic decay. There was good agreement between the isotopic amounts of U and Pu measured by IDMS and those calculated from the weights of the starting materials and the final solution.

Aliquots of the solution were subsequently dispensed into penicillin-type vials.

Aliquoting of batch solution

The solution in the flask was re-weighed and adjusted for the small evaporation losses during the time the verification of the batch measurements were done.

Aliquots of the solution of about 2.5 g were transferred into penicillin-type vials using a commercial, manually operated dispenser. Prior to dispensing, the vials were pre-engraved with the reference material name (IRMM-1027j) and an individual running number starting at 0001. Each vial contained ca. 1.8 mg Pu and 50 mg U. Metrological weighings were carried out on an analytical balance (Mettler AT261 Delta range). The amount dispensed into the vial was measured by tarring the empty vial and weighing after adding the solution. The evaporation effect during aliquoting was also measured for the first ten samples by using a syringe to dispense the aliquots, weighing it before and after dispensing. Subsequent aliquots, directly weighed into the vials were corrected for the small evaporation losses measured on the first 10 samples. A standard weight of 2.5 g with an empty vial was weighed after every 48th aliquot in order to check the long-term stability of the balance.(to be confirmed/checked by Roger)

The LSD spike solution was weighed into the vials over a period of about 14 days. Batches of 24 vials were prepared and kept in a Perspex holder that fitted into a plastic box and each box was closed and stacked with the others ready for drying. By this procedure, 1205 vials were filled over a period of 11 days. A series of witness samples was taken consisting of a daily sample, one at the beginning, one at the end over various days during the weighing campaign (10 samples in total). These witness samples were stored separately. The boxes with the penicillin vials were transferred into one of the drying boxes for the next processes: drying and covering with CAB.

Drying solutions and addition and drying of CAB

The solutions were dried by gentle heating on a thermostatically controlled hot-plate at approx. 55° C. When the solutions had dried (typically 4-5 days continuous heating), about 0.25 mL of a 10% cellulose acetate butyrate (CAB) solution in acetone was added, the solution allowed to evaporate at room temperature for two hours and then heated at approx. 50° C for up to two hours to dry completely.

Two separate glove-boxes were used for the drying allowing up to 48 samples per week to be dried and covered with CAB. The vials containing dried samples were stacked horizontally and inspected regularly. If the material appeared to have flowed even slightly in the vial the vial was heated again to remove the last traces of solvent. The vials containing the dried material covered with CAB were closed with an iso-versilic stopper and an aluminium cap. The vials were then labelled and sealed in PVC packages for storage.

Drying, coating with CAB layer and packing were carried out over a period of several months.

Verification of U and Pu amount content in selected vials

After drying and CAB covering were complete, six vials were chosen at random for verification measurements. To each of these, 5 g of IRMM-046b spike was weighed in and the standard IDMS procedure and the working instructions above were used for the measurement of U and Pu amount content in the spikes. The uranium isotopic ratios $n(^{238}U)/n(^{233}U)$ were measured on the Triton, using the same procedure that was used for the batch solution verifications and the plutonium ratios $n(^{239}Pu)/n(^{242}Pu)$ were measured on the MAT 262.

The results of the verification measurements described above are given in Table 3 and Table 4 and shown in Figs. 1 and 2. These measurements gave values that agreed well with the values for uranium and plutonium amount content calculated from the amounts of dissolved metals and solution.

Table 3: Amount content of uranium. Values from certificates and metrological weighing are compared with values calculated from measurement of samples of the batch solution and from vials. Expanded Uncertainties are given in brackets (coverage factor *k*=2).

	Certificate	Batch	Vials
C(U) mol·g^{-1}	8.088 9(17) · 10^{-5}	8.079(14) · 10^{-5}	8.081(13) · 10^{-5}

Table 4: Amount content of plutonium. Values from the certificate and metrological weighing are compared with values calculated from measurement of samples of the batch solution and from vials. Expanded Uncertainties are given in brackets (coverage factor *k*=2).

	Certificate	Batch	Vials
C(Pu) mol·g^{-1}	3.079 2(14) · 10^{-6}	3.075 5(46) · 10^{-6}	3.075 5(27) · 10^{-6}

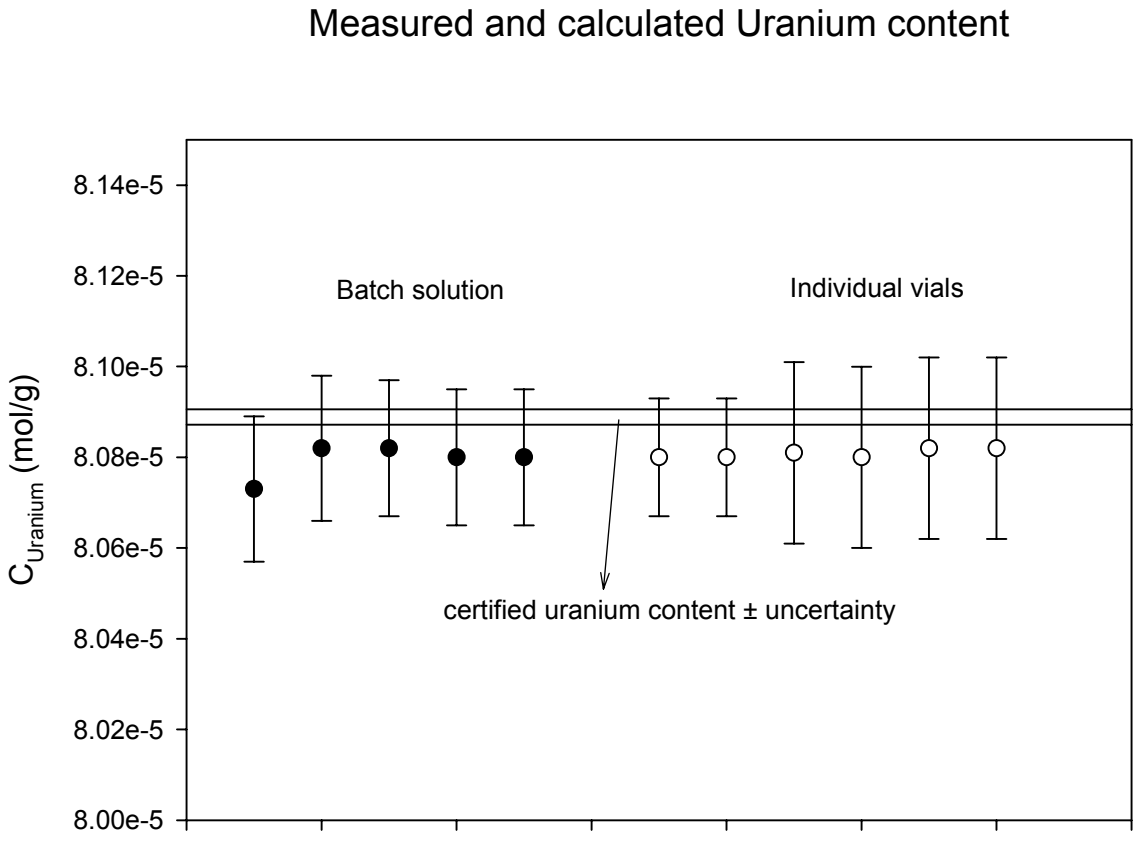


Figure 1: 'Metrological' concentration of uranium in IRMM-1027j (from the weights of metals and solution) compared with the measured values by IDMS.

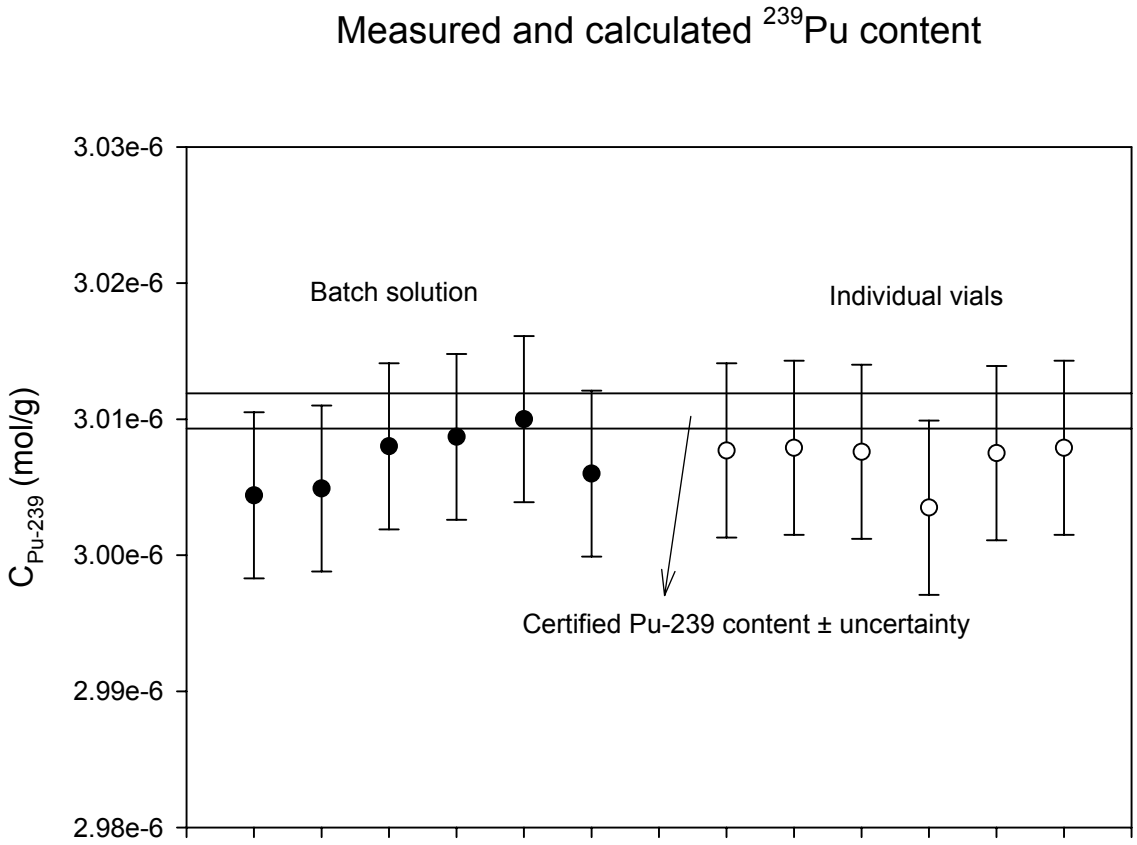


Figure 2: 'Metrological' concentration of plutonium in IRMM-1027j (from the weights of metals and solution) compared with the measured values by IDMS.

Conclusion

A new series of LSD spikes for IDMS determinations of uranium and plutonium contents in solutions of spent nuclear fuel from reprocessing plants has been prepared.

The certification of the spike is based on the metrological data, the certificate of the base materials and the verification measurements. The final certification values are established by mass-metrology of the metals and the solutions.

The verification of the certified values from the mass-metrology was accomplished by IDMS measurements on the batch solution and individual vials. The agreement was satisfactory.

The materials prepared are commercially available from IRMM, Geel as reference material IRMM-1027j for application in the nuclear safeguards measurements of uranium and plutonium in input solutions.

References

[1] Preparation and Certification of a new Type of Large Size Dried Spikes, Batch IRMM-1027f, A Alonso, R Eykens, F Kehoe, H Kühn, N Surugaya, A Verbruggen, R. Wellum, GE/R/IM/36/02

[2] New Procedures for Uranium Isotope Ratio Measurements using the new TRITON Thermal Ionisation Mass Spectrometer, S. Richter, A. Alonso, H. Kühn, R. Wellum, P.D.P. Taylor, Report EUR 21849

Annex 1: Certificate of uranium metal: EC NRM-101

Certified Nuclear Reference Material

Certificate of Analysis

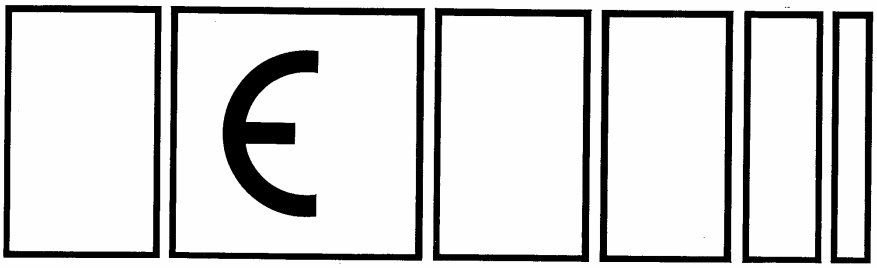
EC NUCLEAR REFERENCE MATERIAL NO. 101

MATERIAL : URANIUM METAL

URANIUM MASS FRACTION : (999.85 ± 0.05) g·kg^{-1}

The uncertainty has been calculated by multiplying the estimated overall standard deviation by a factor of two. This corresponds to a confidence level of about 95 percent.

Commission of the European Communities
Joint Research Centre
Geel Establishment (CBNM)



Annex 2: Certificate of uranium metal: NBL CRM-116

U. S. Department of Energy
New Brunswick Laboratory

New Brunswick Laboratory
Certified Reference Materials
Certificate of Analysis

CRM 116

Uranium (Enriched) Metal
(Uranium and Uranium-235 Standard)

Uranium (etched metal basis) ..... $99.967_2 \pm 0.006_9$ Wt.%
($\alpha = 0.05$, n = 6)

Uranium-235 ..... $93.121_5 \pm 0.004_7$ Wt.%
($\alpha = 0.05$, n = 6)
$93.183_7 \pm 0.004_7$ At.%

Relative atomic weight 235.201

Metal must be etched in 1 + 1 HNO_3, rinsed in distilled-deionized water and acetone, and dried prior to use.

REFERENCE METHODS OF ANALYSIS: Titrimetry (high precision NBL method) verified with NBL CRM 112-A Uranium Metal Standard and thermal ionization mass spectrometry verified with NBL CRM U930 Uranium Isotopic Standard.

June 1978
Argonne, Illinois

Carleton D. Bingham
Director

Annex 3: Certificate of plutonium metal: Cetama MP2

CETAMA

COMMISSARIAT A L'ENERGIE ATOMIQUE

COMMISSION D'ETABLISSEMENT DES METHODES D'ANALYSE

cea

REFERENCE MATERIAL CERTIFICATE

PLUTONIUM METAL

"MP2"

Sample n° Axxx Mass : 0.xxxxxx ± 0.000012 g

(For the values x see page 4)

The reference material to which this certificate relates is intended for the calibration of chemical composition measurement. The overall chemical content of plutonium is certified. The confidence interval associated with the certified value for a single sample, takes into account uncertainties associated to with analysis and heteregenity of metal. This content, expressed as a percentage of mass, was the following on 12 march 2002 for a single sample with a probability level of 0.95.

99.90 ± 0.04 %

THE TRUE MASS OF THE SAMPLE A ± **12 µg**, RELATED TO A VACUUM, IS THAT INDICATED IN THIS CERTIFICATE AND ON THE AMPOULE.

The possibility of surface oxidation makes it impossible to envisage weighing at the time of use

Isotopique composition is certified on 12 march 2001 : see certificate IRMM page3

The preparation, analysis and certification of the plutonium to which this certificate relates was carried out by different units of the CEA group under the supervision of the Committee for Establishing Analysis Methods (CETAMA).

Le responsable MR

CETAMA
certifié
ISO 9001:2000
par

CETAMA
CEA VALRHO Marcoule
B. P. 17171
30207 BAGNOLS SUR CEZE CEDEX FRANCE
Téléphone (33) 4.66.79.69.88 - Télécopie (33) 4.66.79.69.89

Version : 06/2001

On 12/03/200, the metal contained around:

- by weight, 489 $mg.kg^{-1}$ of uranium,
- by weight, 438 $mg.kg^{-1}$ of américium..

UTILISATION

The sample, which consists of a piece of metal, is supplied in a double glass ampoule filled with pure nitrogen at a pressure of around 0.1 Pascal.

The ampoule must be opened with care inside a glove box. All the sample must be transferred to the dissolver.

Cover with 0.1 $mol.l^{-1}$ hydrochloric acid. The ampoule must be thoroughly washed with the same acid to recover any particles of metal which may have become separated. In 2 ml fractions, add the necessary quantity of 12 $mol.l^{-1}$ hydrochloric acid of guaranteed purity to obtain a 4 $mol.l^{-1}$ hydrochloric acid solution. Allow dissolving to proceed without heating for 10 to 15 minutes, then heat to boiling point. If there are still particles of plutonium at the bottom of the dissolver after heating for two hours, add 2 ml of 12 $mol.l^{-1}$ hydrochloric acid and 2 drops of 1 $mol.l^{-1}$ hydrofluoric acid and continue heating for another two hours. Repeat the operation if necessary until the material is totally dissolved.
If plutonium fluoride precipitates out, add a few drops of aluminium nitrate (approximately one $mol.l^{-1}$)..
Allow to cool and adjust to the required volume.

ADDITIONAL INFORMATION

The certified plutonium content has been deduced from analysis of impurities carried out by five laboratories and checked by chemical assay of the plutonium in two different laboratories using three different methods of analysis.
Spark Source Mass Spectrometry has given a full analysis of the impurities and, where concentration levels allowed, inductively-coupled plasma atomic emission spectrometry has been used to establish the concentrations of some of them.
The uranium was determined by laser spectrofluorimetry and the americium by gamma spectrometry.Carbon was determined by coulometry, after transformation into gaseous form by combustion in oxygen.
The gases were analysed by chromatography in the aqueous phase:

- for nitrogen and oxygen after extraction by high temperature stream under an inert gas,
- for hydrogen after diffusion in a vacuum.

Annex 4: Certificate of plutonium metal: isotopic abundances IRMM

EUROPEAN COMMISSION
DIRECTORATE GENERAL JRC
JOINT RESEARCH CENTRE
IRMM
Institute for Reference Materials and Measurements

CERTIFICATE of a reference measurement

IM/MeaC/07/116

11 April 2007

SUBJECT : Recertification of CEA CETAMA MP2

1. Applicant: A. Verbruggen

2. Sample Identification:
   - CEA/CETAMA/MP2
   - Chemical form: Pu metal provided by CEA/CETAMA

3. Measurands:
   - Isotopic composition

isotope amount ratio(s)	
$n(^{238}Pu)/\ n(^{239}Pu)$	0.000 030 83(29)
$n(^{240}Pu)/\ n(^{239}Pu)$	0.022 432 4(51)
$n(^{241}Pu)/\ n(^{239}Pu)$	0.000 237 8(31)
$n(^{242}Pu)/\ n(^{239}Pu)$	0.000 075 70(78)

amount fraction (·100)		mass fraction (·100)	
$n(^{238}Pu)/n(Pu)$	0.003 015(29)	$m(^{238}Pu)/m(Pu)$	0.003 002(28)
$n(^{239}Pu)/n(Pu)$	97.773 05(58)	$m(^{239}Pu)/m(Pu)$	97.763 80(59)
$n(^{240}Pu)/n(Pu)$	2.193 28(49)	$m(^{240}Pu)/m(Pu)$	2.202 27(49)
$n(^{241}Pu)/n(Pu)$	0.023 25(30)	$m(^{241}Pu)/m(Pu)$	0.023 44(31)
$n(^{242}Pu)/n(Pu)$	0.007 402(76)	$m(^{242}Pu)/m(Pu)$	0.007 494(77)

molar mass: 239.074 790 8(91) $g \cdot mol^{-1}$

4. Date of sample receipt : n.a.
   Date of completion of measurement : 7 November 2006

5. All uncertainties indicated are expanded uncertainties $U = k \cdot u_c$ where u_c is the combined standard uncertainty estimated following the ISO/BIPM guide[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. The primary certified values are the isotope amount ratio ; other values are derived from them. Reproducing the derived values may result in difference due to rounding errors.

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

Uncertainty budget :

Quantity	Value	Standard Uncertainty	Index
Atomic mass ^{239}Pu	239.05215760 g/mol	$5.1 \cdot 10^{-6}$ g/mol	59.6 %
Measurement ratio 240/239	0.02243535 mol/mol	$3.81 \cdot 10^{-6}$ mol/mol	14.9 %
Measurement ratio 241/239	$240 \cdot 10^{-6}$ mol/mol	$450 \cdot 10^{-9}$ mol/mol	0.9 %
Measurement ratio 242/239	$75 \cdot 10^{-6}$ mol/mol	$175 \cdot 10^{-9}$ mol/mol	0.4 %
$variability_{241/239}$	0.0 mol/mol	$2.65 \cdot 10^{-6}$ mol/mol	21.0 %
$variability_{242/239}$	0.0 mol/mol	$650 \cdot 10^{-9}$ mol/mol	3.0 %
M_{Pu}	239.07478500 g/mol	$6.46 \cdot 10^{-6}$ g/mol	

6. The traceability to SI is established through standards from IRMM-290.

7. Analytical measurement procedure
   - Mass spectrometric measurments were performed by H Kühn an F Kehoe for the $[n(^{238}Pu)/n(^{239}Pu)]$, $[n(^{240}Pu)/n(^{239}Pu)]$, $[n(^{241}Pu)/n(^{239}Pu)]$ and $[n(^{242}Pu)/n(^{239}Pu)]$ using the MAT262 TIMS, sample solutions were prepared for TIMS analysis by F Kehoe. A. Verbruggen was responsible for preparation and issuance of the certificate.
   - The atomic masses, used in the calculation are from G. Audi and A.H. Wapstra.[2]
   - Reference numbers of the measurement data: measurements number T26629, T26A03, T26B07, logged in S:\D04-IM\Secure Data\Project Data\MP2 (based on 081a and LSD1027i)\MP2 IA Summary MAT262 measurements.
   - Full details of the preparation and the certification procedure can be found in certification report EUR*****.

8. These samples will be stored for a minimum period of six months from the date of this certificate

André Verbruggen
Group leader Nuclear Chemistry

Stephan Richter
Group leader Nuclear Mass Spectrometry

Copies
P Taylor, IM unit head
Y Aregbe, Action leader Nuclear Safeguards
F Kehoe
H Kühn

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

Annex 5: Mass Metrology certificate: base materials

JOINT RESEARCH CENTRE
EUROPEAN COMMISSION

Certificate of weighing

Institute for Reference Materials and Measurements

E. 3514 **Issued date**: 08 November 2005 **Page** 1 **of** 1

Applicant: Mr Verbruggen **Group: IM-Nuclear**

Project: 1027 J

Description: Uranium and plutonium metals.

Date of receipt of request: 30 June 2005 **Weighing date**: 24 July 2005
03 November2005

The reported results applies only to the objects / samples described in this certificate

Mass Pu MP2 (BC 02701) in g.	Mass U EC 101 in g (BC 00626).	Mass U NBL CRM-116 in g. (BC 02158)
2.2872 (2)	47.427 (4)	12.181 (1)

Observations:
The measurements and uncertainty estimates, were performed according to working instruction WI-0185, "Mass determination by substitution weighing"on balance AT 21 comparator and AT 201. The reported weighing result is valid when the air density is 1.20 ±0.03 kgm^{-3} and the object density is 1000 ±100 kgm^{-3}.

Traceability:
The certified mass values are traceable to the International Kilogram Prototype via regular calibrations of the IRMM principal kilogram. The set of working mass standards M10 and M3 were used as reference in the mass determination.

Uncertainty:
All reported uncertainties are expanded uncertainties $U = k \cdot u_c$ where u_c is the combined standard uncertainty calculated according to the ISO/BIPM Guide to the expression of Uncertainty in Measurement. The coverage factor $k = 2$ corresponds to a coverage probability of about 95%. U applies to the last digit of the value of the measurement result and is given in parentheses ().

Annexes:

Signature
Mass Metrology Service

Retieseweg, B-2440 Geel, Belgium; Tel.: +32-(0)14-571 211 • Fax: +32-(0)14-571 978• http://www.irmm.jrc.be

Annex 6: Mass Metrology certificate: primary solution

EUROPEAN COMMISSION DIRECTORATE GENERAL Joint Research Centre	**Certificate of weighing**	Institute for Reference Materials and Measurements

E. 3636 **Issued date**: 3 July 07 **Page** 1 **of** 1

Applicant: Verbruggen **Group:** IM-Nuclear

Project: 1027 J **IM-unit ref.:**

Description: Preparation of 1027 J mother solution

Date of receipt of request: 30 June 2005 **Weighing date**: 24 June 2006

The reported results applies only to the objects / samples described in this certificate

Mass 1027 J mother solution in g: 3102.90 (5)

Observations:

The measurements and uncertainty estimates, were performed according to working instruction WI-0185, "Mass determination by substitution weighing"on balances PR 5002 with IRMM inventory No 1997 00368 28.

Traceability:

The certified mass values are traceable to the International Kilogram Prototype via regular calibrations of the IRMM principal kilogram. The set of working mass standards M 10, M 3 and B 2799 was used as reference in the mass determination.

Uncertainty:

All reported uncertainties are expanded uncertainties $U = k \cdot u_c$ where u_c is the combined standard uncertainty calculated according to the ISO/BIPM Guide to the expression of Uncertainty in Measurement. The coverage factor $k = 2$ corresponds to a coverage probability of about 95%. U applies to the last digit of the value of the measurement result and is given in parentheses ().

Annexes:

.....
Signature
Mass Metrology Service

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Annex 7: Certificate of IRMM-1027j

IRMM

Institute for Reference Materials and Measurements

CERTIFICATE
SPIKE ISOTOPIC REFERENCE MATERIAL IRMM-1027j

This Spike Isotopic Reference Material consists of a certified mass of approximately 2.5 g of solution subsequently evaporated to dryness and covered with a dry layer of circa 50 mg cellulose acetate butyrate to ensure spike integrity.

Each unit is identified by a vial number. The sample mass of the solution for each vial is listed in table 1.

The Isotopic Reference Material (Spike) is supplied with amount concentrations of ^{235}U, ^{238}U and ^{239}Pu certified to be

1.601 30(53) 10^{-5} mol (^{235}U) · g^{-1} (solution)
6.463 7(14) 10^{-5} mol (^{238}U) · g^{-1} (solution)
3.010 6(14) 10^{-6} mol (^{239}Pu) · g^{-1} (solution)

Other uranium and plutonium isotopes present are related to the ^{238}U and ^{239}Pu concentration through the following certified amount ratios:

$n(^{234}U)/n(^{238}U)$	:	0.002 623 8(12)
$n(^{235}U)/n(^{238}U)$	:	0.247 738(80)
$n(^{236}U)/n(^{238}U)$	:	0.001 082 03(68)

$n(^{238}Pu)/n(^{239}Pu)$	:	0.000 030 83(29)
$n(^{240}Pu)/n(^{239}Pu)$	:	0.022 432 4(51)
$n(^{241}Pu)/n(^{239}Pu)$	:	0.000 237 8(31)
$n(^{242}Pu)/n(^{239}Pu)$	:	0.000 075 70(78)

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This corresponds to isotopic compositions of uranium and plutonium with the following abundances :

	amount fraction (·100)		mass fraction (·100)
$n(^{234}U)/n(U)$	0.209 662(81)	$m(^{234}U)/m(U)$	0.206 656(80)
$n(^{235}U)/n(U)$	19.796 2(51)	$m(^{235}U)/m(U)$	19.596 0(51)
$n(^{236}U)/n(U)$	0.086 463(52)	$m(^{236}U)/m(U)$	0.085 953(52)
$n(^{238}U)/n(U)$	79.907 7(52)	$m(^{238}U)/m(U)$	80.111 4(52)

	amount fraction (·100)		mass fraction (·100)
$n(^{238}Pu)/n(Pu)$	0.003 015(29)	$m(^{238}Pu)/m(Pu)$	0.003 002(28)
$n(^{239}Pu)/n(Pu)$	97.773 05(58)	$m(^{239}Pu)/m(Pu)$	97.763 80(59)
$n(^{240}Pu)/n(Pu)$	2.193 28(49)	$m(^{240}Pu)/m(Pu)$	2.202 27(49)
$n(^{241}Pu)/n(Pu)$	0.023 25(30)	$m(^{241}Pu)/m(Pu)$	0.023 44(31)
$n(^{242}Pu)/n(Pu)$	0.007 402(76)	$m(^{242}Pu)/m(Pu)$	0.007 494(77)

The molar mass of the uranium in this sample is 237.445 40(16) $g \cdot mol^{-1}$
The molar mass of the plutonium in this sample is 239.074 790 8(91) $g \cdot mol^{-1}$

From the certified values, the following amount contents are derived:

$8.088\ 9(17) \cdot 10^{-5}$	mol (U) · g^{-1} (solution)
$3.763\ 8(12) \cdot 10^{-3}$	g (^{235}U) · g^{-1} (solution)
$15.386\ 9(33) \cdot 10^{-3}$	g (^{238}U) · g^{-1} (solution)
$19.206\ 8(40) \cdot 10^{-3}$	g (U) · g^{-1} (solution)

$3.079\ 2(14) \cdot 10^{-6}$	mol (Pu) · g^{-1} (solution)
$7.197\ 0(33) \cdot 10^{-4}$	g (^{239}Pu) · g^{-1} (solution)
$7.361\ 6(33) \cdot 10^{-4}$	g (Pu) · g^{-1} (solution)

NOTES

1. This Spike Isotopic Reference Material is traceable to the SI in the shortest possible way. The values of the U and Pu isotope ratios were measured at IRMM and are traceable to the SI via the values of the isotope ratios of the isotopic reference materials IRMM-183, 184, 185, 186, 187 for uranium and IRMM-290 for plutonium. The U and Pu content of this spike are traceable to the SI via reference materials NBL CRM-116, EC NRM-101 and CETAMA MP2. Measurements calibrated by this Isotopic Reference Material have therefore the potential of being traceable to the SI.

2. All uncertainties indicated in this certificate are expanded uncertainties $U = k \cdot u_c$ where u_c is the combined standard uncertainty estimated following the ISO/BIPM Guide to the Expression of Uncertainty in Measurement. They are given in parentheses and include a coverage factor k=2. They apply to the last two digits of the value.

3. The IRMM-1027j was prepared by metrological weighing of U metals (NBL CRM 116, EC NRM 101) and Pu metal (CETAMA MP2), dissolution in HNO_3, subsequently dispensing by metrological weighing into individual units, drying and conditioning in cellulose acetate butyrate (CAB).

4. IRMM-1027j is delivered in individual glass (penicillin) vials each containing about 45 - 50 mg U and 1.8 mg Pu.

5. Values for isotope amount ratios, isotopic compositions and concentrations are valid for 01 January 2007.

6. The half lives used in the calculations are

^{238}Pu : 8.77 (03) $\cdot 10^1$ a[1]
^{239}Pu : 2.411 (03) $\cdot 10^4$ a[2]
^{240}Pu : 6.563 (07) $\cdot 10^3$ a[2]
^{241}Pu : 1.429 (06) $\cdot 10^1$ a[2]
^{242}Pu : 3.735 (11) $\cdot 10^5$ a[2]
^{244}Pu : 8.00 (09) $\cdot 10^7$ a[2]

7. The molar masses, used in the calculations, are[3]

^{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}

^{238}Pu : 238.049 559 9 (40) g·mol^{-1}
^{239}Pu : 239.052 163 4 (40) g·mol^{-1}
^{240}Pu : 240.053 813 5 (40) g·mol^{-1}
^{241}Pu : 241.056 851 5 (40) g·mol^{-1}
^{242}Pu : 242.058 742 6 (40) g·mol^{-1}
^{244}Pu : 244.064 204 (10) g·mol^{-1}

8. The vials should be handled with great care and by experienced personnel in a laboratory environment suitably equipped for the safe handling of radioactive materials.

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[1] IAEA, Decay data of the Transactinium Nuclides, Technical Reports Series No. 261, 1986
[2] P. De Bièvre, A. Verbruggen, 'A new measurement of the ^{241}Pu half-life by isotope mass spectrometry', Int. Conf. on Nuclear Data for Science and Technology, May 19-24, 1997 Trieste, Italy
[3] G. Audi and A.H. Wapstra, The 2003 atomic mass evaluation , Nucl Phys A729 (2003) 337-676.

9. Full details of the certification procedure can be found in the Preparation and Certification Report[4].

Chemical preparation and ampuling of this IRM were accomplished by F Kehoe and R Eykens.

The isotopic verification measurements were carried out by F Kehoe, A Alonso Muñoz, S Richter and H Kühn for uranium and plutonium on samples chemically prepared by F Kehoe and A Alonso Muñoz. Measurements of isotopic ratios were calibrated against synthetic isotopic mixtures prepared by W Lycke for uranium and J Broothaerts for plutonium.

Metrological weighings required in the preparation and certification were performed by F Kehoe and R Eykens.

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

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August 2007

Y Aregbe
IRMM Safeguards Coordinator

P Taylor
Head
Isotope Measurements Unit

[4] A. Verbruggen, A. Alonso, R. Eykens, F. Kehoe, H. Kühn, S. Richter, Y. Aregbe, Preparation and Certification of IRMM-1027j, Large-Sized Dried (LSD) spike, report EUR***** EN

Table 1: list of vial numbers, mass of solution before drying

N°	Mass (g)	N°	Mass (g)	N°	Mass (g)	N°	Mass (g)	N°	Mass (g)	N°	Mass (g)	N°	Mass (g)
1	2.5050	51	2.5554	101	2.5160	151	2.5364	201	2.5386	251	2.5301	301	2.5715
2	2.5103	52	2.6307	102	2.5136	152	2.5260	202	2.5326	252	2.5294	302	2.4987
3	2.5485	53	2.5547	103	2.5202	153	2.5343	203	2.5342	253	2.5215	303	2.5101
4	2.6009	54	2.6342	104	2.5150	154	2.5375	204	2.5357	254	2.5291	304	2.5036
5	2.5178	55	2.5572	105	2.5188	155	2.5250	205	2.5380	255	2.5285	305	2.5291
6	2.5051	56	2.5932	106	2.5165	156	2.5282	206	2.5339	256	2.5327	306	2.5146
7	2.5068	57	2.5836	107	2.5186	157	2.5343	207	2.5304	257	2.5270	307	2.5174
8	2.5368	58	2.6381	108	2.5186	158	2.5322	208	2.5385	258	2.5309	308	2.5082
9	2.4936	59	2.6470	109	2.4758	159	2.5276	209	2.5306	259	2.5276	309	2.5165
10	2.4644	60	2.6362	110	2.5131	160	2.5271	210	2.5319	260	2.5313	310	2.5167
11	2.4943	61	2.6535	111	2.4745	161	2.5287	211	2.5302	261	2.5353	311	2.5221
12	2.4700	62	2.5237	112	2.5280	162	2.5287	212	2.5350	262	2.5296	312	2.5389
13	2.6353	63	2.5171	113	2.5336	163	2.5306	213	2.5294	263	2.5290	313	2.5356
14	2.6269	64	2.5233	114	2.5274	164	2.5264	214	2.5324	264	2.5275	314	2.4919
15	2.6004	65	2.5112	115	2.5323	165	2.5295	215	2.5329	265	2.5236	315	2.5250
16	2.5629	66	2.5069	116	2.5300	166	2.5331	216	2.5300	266	2.5338	316	2.5399
17	2.6057	67	2.5114	117	2.5345	167	2.5302	217	2.5301	267	2.5302	317	2.6403
18	2.6255	68	2.5134	118	2.5366	168	2.5357	218	2.5340	268	2.5369	318	2.5399
19	2.6239	69	2.5135	119	2.5333	169	2.5296	219	2.5355	269	2.4898	319	2.5602
20	2.4655	70	2.5196	120	2.5054	170	2.5246	220	2.5363	270	2.5116	320	2.5224
21	2.4913	71	2.5094	121	2.5295	171	2.5377	221	2.5320	271	2.4936	321	2.5336
22	2.4763	72	2.5110	122	2.5410	172	2.5302	222	2.5333	272	2.5268	322	2.5098
23	2.4729	73	-	123	2.5307	173	2.5389	223	2.5326	273	2.5126	323	2.5032
24	2.4983	74	2.5200	124	2.5311	174	2.5303	224	2.5378	274	2.5158	324	2.5114
25	2.4766	75	2.5159	125	2.5319	175	2.5351	225	2.5319	275	3.1666	325	2.5406
26	2.4697	76	2.5068	126	2.5297	176	2.5343	226	2.5340	276	2.5167	326	2.5268
27	2.5364	77	2.5148	127	2.5314	177	2.5342	227	2.5351	277	2.5454	327	2.4921
28	2.4900	78	2.5088	128	2.5325	178	2.5351	228	2.5335	278	2.4744	328	2.5528
29	2.5255	79	2.5120	129	2.5291	179	2.5334	229	2.5348	279	2.5617	329	2.5159
30	2.4827	80	2.5163	130	2.5300	180	2.5360	230	2.5307	280	2.6235	330	2.5363
31	2.5723	81	2.5123	131	2.5292	181	2.5350	231	2.5389	281	2.5099	331	2.5243
32	2.5712	82	2.5100	132	2.5324	182	2.5333	232	2.5331	282	2.5083	332	2.5109
33	2.5322	83	2.5207	133	2.5288	183	2.5336	233	2.5330	283	2.5042	333	2.5099
34	2.5326	84	2.5167	134	2.5364	184	2.5312	234	2.5373	284	2.5230	334	2.5393
35	2.4747	85	2.5145	135	2.5344	185	2.5336	235	2.5318	285	2.5087	335	2.5267
36	2.5486	86	2.5119	136	2.5345	186	2.5320	236	2.5341	286	2.5144	336	2.5223
37	2.5350	87	2.5122	137	2.5310	187	2.5287	237	2.5267	287	2.5289	337	2.5411
38	2.5292	88	2.5107	138	2.5317	188	2.5301	238	2.5269	288	2.5093	338	2.5280
39	2.5176	89	2.5210	139	2.5325	189	2.5299	239	2.5270	289	2.5140	339	2.5280
40	2.4980	90	2.5131	140	2.5273	190	2.5280	240	2.5321	290	2.5018	340	2.5189
41	2.4977	91	2.5086	141	2.5271	191	2.5296	241	2.5330	291	2.5418	341	2.5042
42	2.5555	92	2.5101	142	2.5291	192	2.5327	242	2.5255	292	2.4949	342	2.4978
43	2.5249	93	2.5120	143	2.5308	193	2.5356	243	2.5279	293	2.5091	343	2.5484
44	2.5068	94	2.5195	144	2.5298	194	2.5348	244	2.5314	294	2.5227	344	2.5476
45	2.4870	95	2.5070	145	2.5297	195	2.5376	245	2.5270	295	2.5186	345	2.5195
46	2.5080	96	2.5053	146	2.5306	196	2.5407	246	2.5302	296	2.5278	346	2.5209
47	2.5192	97	2.5155	147	2.5387	197	2.5338	247	2.5280	297	2.5325	347	2.4644
48	2.4872	98	2.5128	148	2.5354	198	2.5340	248	2.5253	298	2.5867	348	2.5210
49	2.4565	99	2.5157	149	2.5364	199	2.5377	249	2.5338	299	2.5016	349	2.5481
50	2.5881	100	2.5117	150	2.5365	200	2.5397	250	2.5272	300	2.5244	350	2.5249

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Table 1: list of vial numbers, mass of solution before drying (continued)

N°	Mass (g)	N°	Mass (g)	N°	Mass (g)	N°	Mass (g)	N°	Mass (g)	N°	Mass (g)	N°	Mass (g)
351	2.5374	401	2.5595	451	2.5022	501	2.5160	551	2.5056	601	2.5437	651	2.5486
352	2.5761	402	2.5606	452	2.5802	502	2.5306	552	2.5300	602	2.5216	652	2.5345
353	2.5168	403	2.5358	453	2.5175	503	2.5279	553	2.5283	603	2.5218	653	2.5172
354	2.5362	404	2.5220	454	2.5933	504	2.5348	554	2.5296	604	2.4970	654	2.5297
355	2.5152	405	2.5330	455	2.5350	505	2.5460	555	2.5431	605	2.5199	655	2.5106
356	2.5144	406	2.5281	456	2.5654	506	2.5357	556	2.5130	606	2.5043	656	2.5262
357	2.5536	407	2.5288	457	2.5883	507	2.5385	557	2.5338	607	2.5158	657	2.5244
358	2.5193	408	2.5396	458	2.5371	508	2.5411	558	2.5088	608	2.5432	658	2.5275
359	2.5099	409	2.5141	459	2.5000	509	2.5115	559	2.5231	609	2.5137	659	2.5107
360	2.5016	410	2.5497	460	2.5236	510	2.5194	560	2.5031	610	2.5154	660	2.5126
361	2.5126	411	2.5262	461	2.5837	511	2.5261	561	2.5661	611	2.5198	661	2.4966
362	2.5410	412	2.5244	462	2.5100	512	2.5056	562	2.5290	612	2.5142	662	2.5255
363	2.5069	413	2.5200	463	2.5173	513	2.5184	563	2.5346	613	2.5416	663	2.5293
364	2.5077	414	2.5546	464	2.4940	514	2.5796	564	2.5175	614	2.5204	664	2.5454
365	2.5559	415	2.5314	465	2.5195	515	2.5505	565	2.5028	615	2.5076	665	2.5152
366	2.5484	416	2.5437	466	2.5067	516	2.5090	566	2.5379	616	2.5278	666	2.5366
367	2.5044	417	2.5274	467	2.5528	517	2.5326	567	2.5260	617	2.4999	667	2.5243
368	2.5769	418	2.5328	468	2.5465	518	2.5260	568	2.5076	618	2.5258	668	2.5034
369	2.5342	419	2.5334	469	2.5668	519	2.5357	569	2.5321	619	2.4977	669	2.5232
370	2.5414	420	2.5019	470	2.5181	520	2.5327	570	2.5300	620	2.5297	670	2.5519
371	2.5098	421	2.5749	471	2.5284	521	2.5284	571	2.5157	621	2.5095	671	2.5026
372	2.5780	422	2.5089	472	2.5246	522	2.5143	572	2.5246	622	2.4976	672	2.5026
373	2.5077	423	2.5644	473	2.5315	523	2.5223	573	2.5341	623	2.5355	673	2.6668
374	2.5267	424	2.5231	474	2.5013	524	2.5108	574	2.5313	624	2.5266	674	2.9346
375	2.5205	425	-	475	2.5551	525	2.5314	575	2.5161	625	2.5400	675	2.5130
376	2.5586	426	2.5374	476	2.5363	526	2.5364	576	2.5368	626	2.5266	676	2.5435
377	2.5467	427	2.5249	477	2.5655	527	2.5279	577	2.5302	627	2.5555	677	2.5124
378	2.5170	428	2.5077	478	2.5158	528	2.5254	578	2.5260	628	2.5082	678	2.5300
379	2.5565	429	2.5000	479	2.5494	529	2.5354	579	2.5276	629	2.5423	679	2.5107
380	2.5236	430	2.5733	480	2.5707	530	2.5342	580	2.5264	630	2.5161	680	2.5467
381	2.4946	431	2.5423	481	2.5760	531	2.4968	581	2.5428	631	2.5033	681	2.5183
382	2.5201	432	2.5031	482	2.5848	532	2.5187	582	2.5403	632	2.5268	682	2.5593
383	2.5096	433	2.5218	483	2.5465	533	2.5205	583	2.5078	633	2.5091	683	2.5274
384	2.5524	434	2.5302	484	2.5881	534	2.5277	584	2.4996	634	2.5083	684	2.5923
385	2.4934	435	2.5072	485	2.5221	535	2.5153	585	2.5109	635	2.5318	685	2.5440
386	2.5062	436	2.5055	486	2.5358	536	2.5323	586	2.5106	636	2.5364	686	2.5119
387	2.5344	437	2.5764	487	2.5427	537	2.5199	587	2.5271	637	2.5342	687	2.5393
388	-	438	2.4974	488	2.5618	538	2.5233	588	2.5160	638	2.4970	688	2.5484
389	2.5457	439	2.5072	489	2.5506	539	2.5074	589	2.5205	639	2.5394	689	2.5056
390	2.4966	440	2.5110	490	2.5309	540	2.5110	590	2.5374	640	2.5147	690	2.5097
391	2.5344	441	2.5028	491	2.4946	541	2.5024	591	2.5201	641	2.5515	691	2.5174
392	2.4912	442	2.5652	492	2.5319	542	2.5304	592	2.5349	642	2.5303	692	2.5559
393	2.5595	443	2.5369	493	2.5350	543	2.5455	593	2.5128	643	2.5551	693	2.5160
394	2.5063	444	2.5185	494	2.5532	544	2.5084	594	2.5039	644	2.5206	694	2.5019
395	2.5586	445	2.5767	495	2.5642	545	2.5507	595	2.5079	645	2.5048	695	2.5304
396	2.5120	446	2.5546	496	2.5409	546	2.5176	596	2.5400	646	2.5276	696	2.5120
397	2.5413	447	2.5386	497	2.5277	547	2.4998	597	2.5046	647	2.5526	697	2.5084
398	2.5262	448	2.5624	498	2.5308	548	2.5359	598	2.5156	648	2.5417	698	2.4992
399	2.5324	449	2.5000	499	2.5115	549	2.5236	599	2.4994	649	2.5949	699	2.5484
400	2.5219	450	2.5771	500	2.5536	550	2.5316	600	2.5612	650	2.5237	700	2.5172

Table 1: list of vial numbers, mass of solution before drying (continued)

N°	Mass (g)	N°	Mass (g)	N°	Mass (g)	N°	Mass (g)	N°	Mass (g)	N°	Mass (g)		Mass (g)
701	2.5718	751	2.5492	801	2.5364	851	2.5261	901	2.5346	951	2.5138	1001	2.5431
702	2.5054	752	2.5358	802	2.5335	852	2.5119	902	2.5088	952	2.5279	1002	2.5174
703	2.5226	753	2.5212	803	2.5256	853	2.5139	903	2.4964	953	2.5231	1003	2.5269
704	2.5437	754	2.5123	804	2.5205	854	2.5052	904	2.5192	954	2.5393	1004	2.5258
705	2.5396	755	2.5361	805	2.5209	855	2.5094	905	2.5261	955	2.5656	1005	2.5328
706	2.5438	756	2.5479	806	2.5106	856	2.5355	906	2.5278	956	2.5428	1006	2.5160
707	2.5440	757	2.5395	807	2.5223	857	2.5397	907	2.5294	957	2.5087	1007	2.5051
708	2.5363	758	2.5246	808	2.4967	858	2.5145	908	2.5584	958	2.5321	1008	2.5258
709	2.5427	759	2.5816	809	2.5384	859	2.5334	909	2.5188	959	2.5573	1009	2.5110
710	2.5723	760	2.5025	810	2.5165	860	2.4954	910	2.5173	960	2.4999	1010	-
711	2.4960	761	2.5156	811	2.5159	861	2.5082	911	2.5238	961	2.5170	1011	2.5301
712	2.5308	762	2.5514	812	2.5247	862	2.5119	912	2.5183	962	2.5044	1012	2.5033
713	2.5565	763	2.5305	813	2.5377	863	2.5556	913	2.5092	963	2.5224	1013	2.5009
714	2.5271	764	2.5240	814	2.5140	864	2.5104	914	2.4986	964	2.5253	1014	2.5267
715	2.4938	765	2.5159	815	2.5160	865	2.5078	915	2.5120	965	2.5296	1015	2.5115
716	2.5400	766	2.5016	816	2.5035	866	2.4934	916	2.5770	966	2.5290	1016	2.5185
717	2.4959	767	2.5274	817	2.5065	867	2.4997	917	2.5223	967	2.5361	1017	2.5254
718	2.5444	768	2.5043	818	2.5561	868	2.5232	918	2.5008	968	2.5332	1018	2.5293
719	2.5818	769	2.5093	819	2.5056	869	2.5034	919	2.5381	969	2.5093	1019	2.5235
720	2.5109	770	2.5326	820	2.5086	870	2.4991	920	2.5316	970	2.5126	1020	2.5578
721	2.5302	771	2.5284	821	2.5290	871	2.5046	921	2.5371	971	2.5336	1021	2.5221
722	2.5091	772	2.5101	822	2.5139	872	2.4956	922	2.5301	972	2.5477	1022	2.5240
723	2.5840	773	2.5415	823	2.5501	873	2.5127	923	2.5194	973	2.5437	1023	2.4938
724	2.5303	774	2.5263	824	2.5394	874	2.5406	924	2.5103	974	2.5192	1024	2.5149
725	2.5658	775	2.5199	825	2.5110	875	2.5155	925	2.5320	975	2.5094	1025	2.4964
726	2.5423	776	2.5045	826	2.5129	876	2.5195	926	2.4976	976	2.5486	1026	2.5114
727	2.5215	777	2.5090	827	2.5389	877	2.5150	927	2.5240	977	2.5249	1027	2.5346
728	2.6201	778	2.5742	828	2.5364	878	2.5504	928	2.5435	978	2.5159	1028	2.5055
729	2.6151	779	2.5586	829	2.5091	879	2.5142	929	2.5003	979	2.5265	1029	2.5116
730	2.5987	780	2.5067	830	2.5232	880	2.5189	930	2.5190	980	2.5577	1030	2.5084
731	2.5129	781	2.5344	831	2.5275	881	2.5227	931	2.5184	981	2.5216	1031	2.5256
732	2.5075	782	2.5033	832	2.5268	882	2.5207	932	2.5240	982	2.5061	1032	2.4941
733	2.5569	783	2.5284	833	2.4996	883	2.5283	933	2.5035	983	-	1033	2.5150
734	2.5228	784	2.5081	834	2.5032	884	2.5137	934	2.5171	984	2.5242	1034	2.6092
735	2.5055	785	2.5151	835	2.5366	885	2.5183	935	2.5180	985	2.5119	1035	2.5404
736	2.4931	786	2.5253	836	2.5124	886	2.5242	936	2.5170	986	2.5274	1036	2.5787
737	2.5041	787	2.5061	837	2.5192	887	2.5168	937	2.5110	987	2.5252	1037	2.4876
738	2.5189	788	2.5089	838	2.5007	888	2.5012	938	2.5248	988	2.5235	1038	2.5015
739	2.5423	789	2.5111	839	2.5268	889	2.5405	939	2.5401	989	2.5325	1039	2.5296
740	2.5315	790	2.5421	840	2.5606	890	2.5143	940	2.4968	990	2.5052	1040	2.4944
741	2.5148	791	2.5184	841	2.5621	891	2.5176	941	2.5200	991	2.5294	1041	2.5350
742	2.5249	792	2.5316	842	2.4990	892	2.5238	942	2.5445	992	2.5296	1042	2.7070
743	2.5153	793	2.4967	843	2.5206	893	2.5484	943	-	993	2.4976	1043	2.7238
744	2.5452	794	2.5282	844	2.5168	894	2.5214	944	2.5400	994	2.5709	1044	2.5041
745	2.5378	795	2.5122	845	2.5003	895	2.5106	945	2.5098	995	2.5274	1045	2.7070
746	2.5374	796	2.5238	846	2.5224	896	2.5513	946	2.5285	996	2.5274	1046	2.8301
747	2.4987	797	2.5033	847	2.5148	897	2.5335	947	2.5096	997	2.5236	1047	2.5344
748	2.5063	798	2.4960	848	2.5076	898	2.5032	948	2.5162	998	2.5327	1048	2.6748
749	2.5375	799	2.5097	849	2.5180	899	2.4963	949	2.5090	999	2.5234	1049	2.5955
750	2.5372	800	2.5191	850	2.5095	900	2.5237	950	2.5228	1000	2.5195	1050	2.6938

N°	Mass (g)	N°	Mass (g)	N°	Mass (g)	N°	Mass (g)
1051	2.5078	1101	2.5774	1151	2.5193	1201	2.5096
1052	2.5090	1102	2.5199	1152	2.5185	1202	2.5043
1053	--	1103	2.5215	1153	2.4972	1203	2.5727
1054	2.5149	1104	2.5094	1154	2.5096	1204	2.6930
1055	2.5069	1105	2.5095	1155	2.6377	1205	2.5169
1056	2.5338	1106	2.5214	1156	2.5241		
1057	2.5181	1107	2.5080	1157	2.5075		
1058	-	1108	2.5029	1158	2.5246		
1059	2.5055	1109	2.5007	1159	2.5152		
1060	-	1110	2.5057	1160	2.5236		
1061	2.5140	1111	2.5309	1161	2.5158		
1062	2.5132	1112	2.5243	1162	2.5092		
1063	2.5366	1113	2.5311	1163	2.5884		
1064	2.5863	1114	2.5216	1164	2.5284		
1065	2.6326	1115	2.5383	1165	2.9790		
1066	2.5389	1116	2.5151	1166	2.5816		
1067	2.5690	1117	2.5133	1167	2.5068		
1068	2.6508	1118	2.5006	1168	2.5027		
1069	2.5079	1119	2.5172	1169	2.5163		
1070	2.5203	1120	2.6202	1170	2.5164		
1071	2.5816	1121	2.5515	1171	2.5096		
1072	2.7534	1122	2.5023	1172	2.4953		
1073	2.6172	1123	2.5291	1173	2.5906		
1074	2.5757	1124	2.5049	1174	2.5098		
1075	2.5426	1125	2.5177	1175	2.5324		
1076	2.5846	1126	2.5281	1176	2.4994		
1077	2.5804	1127	2.5276	1177	2.5096		
1078	2.5142	1128	2.5334	1178	2.5027		
1079	2.5205	1129	2.5267	1179	2.5000		
1080	2.5000	1130	2.5241	1180	2.5067		
1081	2.5013	1131	2.5052	1181	2.5576		
1082	2.5112	1132	2.5181	1182	2.5095		
1083	2.5759	1133	2.5183	1183	2.5134		
1084	2.5379	1134	2.5258	1184	2.5025		
1085	2.5209	1135	2.5109	1185	2.5636		
1086	2.5251	1136	2.5155	1186	2.5019		
1087	2.5090	1137	2.5321	1187	2.5297		
1088	2.5001	1138	2.5021	1188	2.5036		
1089	2.5014	1139	2.5116	1189	2.4974		
1090	2.5078	1140	2.5329	1190	2.5270		
1091	2.4993	1141	2.5055	1191	2.5063		
1092	2.5073	1142	2.5155	1192	2.5030		
1093	2.5299	1143	2.5183	1193	2.4944		
1094	2.5055	1144	2.5138	1194	2.5129		
1095	2.5088	1145	2.5265	1195	2.4995		
1096	2.5154	1146	2.5232	1196	2.5019		
1097	2.5211	1147	2.6093	1197	2.5069		
1098	2.5799	1148	2.5347	1198	2.5019		
1099	2.5200	1149	2.5182	1199	2.5190		
1100	2.6339	1150	2.5234	1200	2.5805		

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Abstract

A new set of Large Size Dried (LSD) Spikes for the determination of uranium and plutonium by isotope dilution mass spectrometry in solutions of spent fuel from reprocessing plants has been prepared and certified for uranium and plutonium isotopic contents. The methodology followed was similar to that of previous batches. The solution, made by dissolution of the starting materials in nitric acid, was dispensed directly into individual penicillin vials.

This new batch of large size dried spikes contains ca. 50 mg of uranium (^{235}U abundance = 19.7%) and ca. 1.8 mg of plutonium (^{239}Pu abundance = 97.8%) in each individual vial, covered with a light layer of organic material (cellulose acetate butyrate) as stabilizer. The U and Pu amount content was certified based on values from mass metrology. Verification of the amount contents of the spike was done by IDMS at IRMM. The values measured for the batch solution and of the dried covered spikes agreed well with those calculated from the weights of starting materials dissolved and the weights of the final solution.

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