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Preparation and Certification of IRMM-1027n, Large-Sized Dried (LSD) Spike

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Institute for Reference Materials and Measurements

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IRMM information
REFERENCE MATERIALS

Preparation and Certification of IRMM-1027n, Large-Sized Dried (LSD) Spike

IRMM-1027n

R. Jakopič, J. Bauwens, T. Drooghmans, R. Eykens, U. Jacobsson, F. Kehoe, H. Kühn, Y. Kushigeta, S. Richter, A. Verbruggen, R. Wellum, Y. Aregbe

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Disclaimer

Certain commercial equipment, instruments, and materials are identified in this report to specify adequately the experimental procedure. In no case does such identification imply recommendation or endorsement by the European Commission, nor does it imply that the material or equipment is necessarily the best available for the purpose.

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Summary

Large-sized dried spikes (LSD) have become a fundamental part of the fissile material control of irradiated nuclear fuel. Within the programme at IRMM to provide these spikes to the nuclear industry and the safeguards' inspectorate, a new set of LSD Spikes for the determination of uranium and plutonium by isotope dilution mass spectrometry (IDMS) in solutions of spent fuel from reprocessing plants has been prepared and certified for uranium and plutonium isotopic contents. The methodology followed was comparable to that of previous batches. The solution, made by dissolution of the starting materials in nitric acid, was dispensed directly into individual penicillin vials. An automated system was used to dispense and weigh the vials.

The new batch of large size dried spikes contains ca. 50 mg of uranium with a ^{235}U amount fraction of 19.5% and ca. 1.8 mg of plutonium with a ^{239}Pu amount fraction of 97.8% in each individual vial, covered with a light layer of organic material (cellulose acetate butyrate) as stabilizer.

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

Introduction

The series IRMM-1027n 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 the 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 and isotope exchange 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% relative amount fraction of ^{235}U, which means for accountability purposes the uranium is classified as 'low enriched'.

As for previous batches of the IRMM 1027 series, high purity metals were chosen as starting materials. It was decided to use CETAMA MP2 plutonium metal and uranium metals EC NRM 101 and CRM-116. This allows the isotopic contents of the LSD spike to be certified from the certificates of the metals (chemical purity and isotopic content), the masses of the metals and the solution.

A single large volume of batch solution is made up; 1200 units are dispensed by the automated system into penicillin vials. The solution in each vial is dried down and then covered with a light organic coating (cellulose acetate butyrate, CAB) dried onto the spike material. 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. Although from experience in the preparation of previous IRMM 1027 series homogeneity has been demonstrated, the isotope amount contents of a selected set of individual spikes after drying are measured by IDMS to confirm the homogeneity of the whole batch. Individual vials are also measured to verify the values for uranium from the mass metrology of the starting metals dissolved and the weight of the final solution and for certification measurements for plutonium.

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 ampoules of MP2 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 that was weighed was that calculated to obtain a solution of ca. 0.7 mg plutonium per gram solution when dissolved in 3 kg nitric acid. The results from the weighing of metal agreed well with the CETAMA certified mass of the MP2 metal.

Uranium metals EC NRM 101, CRM-116

Approximately 47.7 g EC NRM 101 (natural uranium) metal was etched with 1 $mol \cdot L^{-1}$ HNO_3 as recommended on the certificate to remove surface oxides, rinsed with de-ionized 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 11.9 g NBL CRM-116 enriched uranium. The masses of the uranium were calculated so as to yield a solution of ca. 19 mg uranium per gram solution with an enrichment of ca. 19.5% relative amount fraction of ^{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 international kilogram prototype at BIPM, Sèvres. The necessary corrections for air 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. However the plutonium did not dissolve completely as it did in previous batches of the 1027 series. A small amount of a fine white precipitate was observed. Taking into account the limited supply of plutonium metal and that IRMM has a long record of demonstrated measurement capabilities in plutonium analysis, it was decided not to discard the batch solution [1, 2]. Despite incomplete dissolution of plutonium the uranium metals were added to the solution. The uranium dissolved quickly and completely within a few days. The solution was left to stand for another 6 months to homogenize with occasionally swirling by hand and filtered through a separation column filled with about 8 cm of quartz sand (Merck, quartz, fine granular, washed and calcined GR for analysis, SiO_2) to remove the residue. The cleaned solution was collected in a weighed bottle and brought up to the required mass.

Measurement of isotopic abundances in selected vials

The verification of the certified ratios for uranium and plutonium was accomplished by mass spectrometry measurements on a single vial of IRMM-1027n.

The chemical procedure prior to mass spectrometry as detailed in [3] was employed. The purified fractions of U and Pu were prepared in 1 $mol \cdot L^{-1}$ HNO_3 for measurements of the isotopic ratios by TIMS.

The isotopic ratios of the uranium and plutonium were measured on a Thermo Fisher Scientific Triton TIMS following IRMM quality management procedures [4]. The mass spectrometers were calibrated for mass fractionation by measuring IRMM-074/10 uranium and IRMM-290/A3 plutonium isotopic reference material during the procedure.

The measured isotope ratios are compared to the calculated values from the weighing certificates in Table 1 for uranium and in Table 2 for plutonium.

Table 1: Isotopic amount ratios of uranium in a single vial. Values from certificates and metrological weighing are compared with abundances calculated from measurement of isotopic ratios in a single vial. Expanded uncertainties are given in brackets (coverage factor k=2).

	$n(^{234}U)/n(^{238}U)$	$n(^{235}U)/n(^{238}U)$	$n(^{236}U)/n(^{238}U)$
Mass metrology value	0.002 556(10)	0.241 312(72)	0.001 051 5(32)
Measured value	0.002 571(17)	0.241 451(55)	0.001 072(24)

Table 2: Isotopic amount ratios of plutonium in a single vial. Values from certificates and metrological weighing are compared with abundances calculated from measurement of isotopic ratios in a single vial. Expanded uncertainties are given in brackets (coverage factor k=2).

	$n(^{238}Pu)/n(^{239}Pu)$	$n(^{240}Pu)/n(^{239}Pu)$	$n(^{241}Pu)/n(^{239}Pu)$	$n(^{242}Pu)/n(^{239}Pu)$
Mass metrology value	0.000 029 98(28)	0.022 426 3(51)	0.000 200 2(26)	0.000 075 71(78)
Measured value	0.000 038 2(17)	0.022 423 7(16)	0.000 199 90(83)	0.000 076 20(75)

Aliquoting of batch solution

The solution in the flask was re-weighed and adjusted for the small evaporation losses. Prior to dispensing, the vials were cleaned following working instructions "Cleaning of glass penicillin vials for storage of LSD spikes" pre-engraved with the reference material name (IRMM-1027n) and an individual running number starting at 0001.

Automated system aliquoting

The automated system to produce LSD spikes has been installed in collaboration with Nucomat, a company with a recognized reputation in design and development of integrated automated systems. The major components of the system are a robot, two balances, a dispenser and a drying unit fitted into a glove box [5].

The robot is software driven and designed to control all movements inside the glove-box, to identify the penicillin vials with a barcode reader, to dispense the LSD batch solution into the vials and to weigh the amount dispensed. The weighing section is equipped with a semi-analytical balance (Sartorius TE124S) and a 5 kg balance (Sartorius TE6101) to monitor the mass of the mother solution during dispensing and to verify overnight losses by evaporation.

The LSD spike solution was weighed into the vials over a period of 5 days following working instruction "LSD automated system equipment manual". Batches of 48 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. The boxes with the penicillin vials were transferred into one of the drying glove-boxes for the next processes: drying and covering with CAB.

Drying solutions and addition and drying of CAB

The solutions were treated according to the procedure "Processing & Preparation of Large-Size Dried (LSD) Spikes following dispensing" which includes drying by gentle heating on a thermostatically controlled hot-plate at approx. 60 °C. When the solutions had dried (typically 4-5 days continuous heating), about 0.7 mL of a 10% cellulose acetate butyrate (CAB) solution in acetone was added, the solution allowed to evaporate at room temperature for 3 hours and then heated at approx. 45 °C for up to 45 min 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.

Homogeneity of IRMM-1027n batch

Twelve vials were randomly selected to assess the homogeneity of the uranium and plutonium in IRMM-1027n. To each of these, about 5 g of IRMM-046b spike was weighed in, the standard IDMS procedure and the respective instructions were used for the measurement of Pu and U amount content in the spikes. The plutonium isotopic ratios $n(^{239}Pu)/n(^{242}Pu)$ and the uranium isotopic ratios $n(^{235}U)/n(^{233}U)$ were measured on the Triton. During the measurements, one of the vials gave anomalous results for both elements. The relative deviation of these measurement results was of the same order of magnitude and in the same direction for uranium and plutonium, which is a strong indication that the IDMS procedure had failed due to an unobserved slip when adding the U/Pu mixed spike. A further confirmation of this conclusion is that the U/Pu ratio in all 12 measured vials was constant, including the vial giving anomalous results. The results of this vial were therefore discarded. The results for the ^{239}Pu amount content of the single vials described above are shown in Figure 1 for Pu and in Figure 2 for U, together with the mean value of the series.

Figure 1: The amount content of ^{239}Pu in single vials of IRMM-1027n measured by IDMS (with expanded uncertainties, coverage factor k=2).

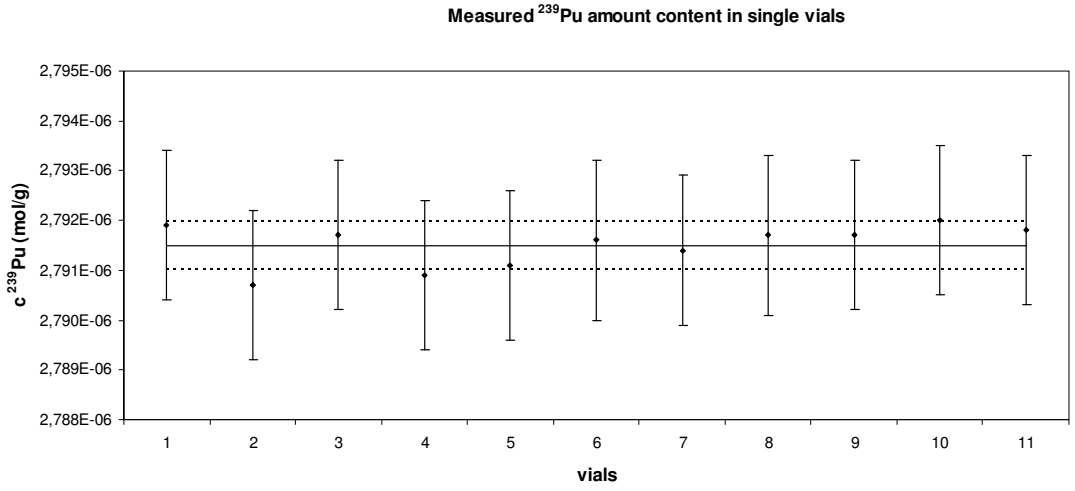
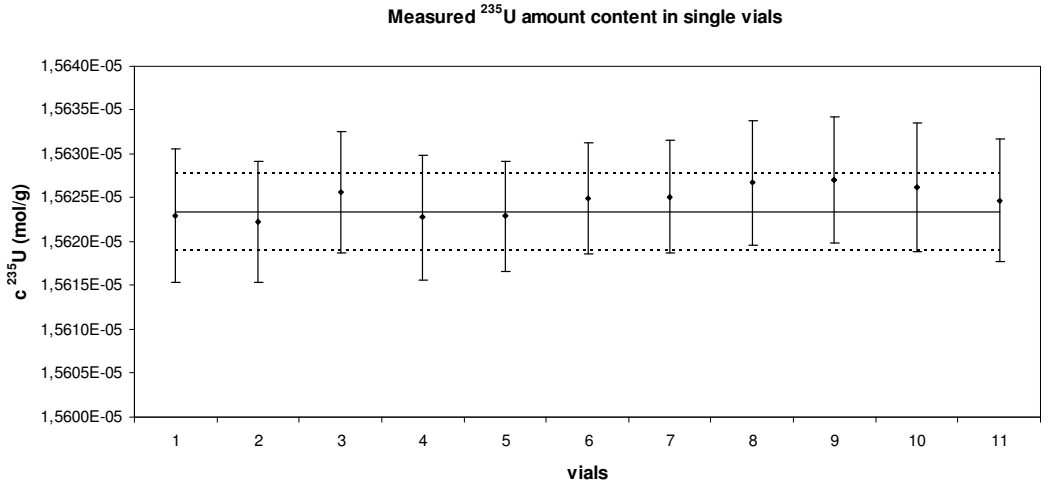


Figure 2: The amount content of ^{235}U in single vials of IRMM-1027n measured by IDMS (with expanded uncertainties, coverage factor k=2).



Certification of Pu amount content in selected vials

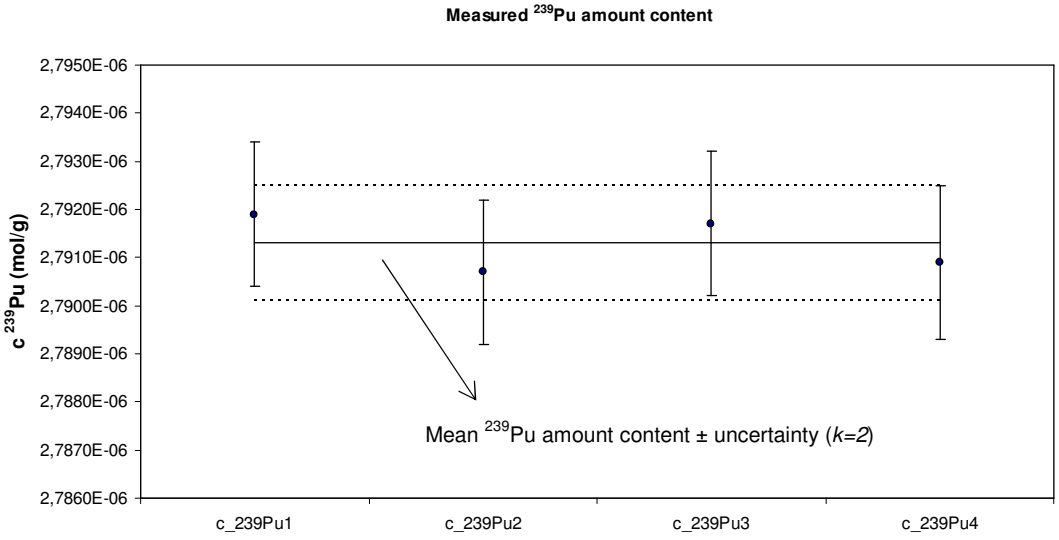
When preparing the 1027n batch solution incomplete dissolution of the MP2 Pu metal was observed. It was decided not to discard the valuable batch solution, but to certify the plutonium amount content by IDMS applying Thermal Ionisation Mass Spectrometry (TIMS).

IDMS is a "primary" reference analytical method which has proven to provide confident and accurate results also in previous batches of the IRMM 1027 series. The IDMS results obtained from the homogeneity study also confirmed the certified uranium and plutonium amount content in IRMM 1027n. Additional verification measurements from other laboratories will be available in the future.

From the eleven measurements described above for the homogeneity measurements, four were chosen at random to provide the certification of the Pu amount content. The certified value for the ^{239}Pu amount content was calculated as the mean value of the certification measurement results and is 2.791 3(12) $\cdot 10^{-6}$ mol$\cdot$g^{-1}. Figure 3 displays the individual IDMS results together with the certified value. The relative expanded uncertainty of the certified value using IDMS is only slightly larger than the relative uncertainty from the metrological weighing of the MP2 Pu metal and is deemed fit for intended use.

The values of the plutonium isotopic content are traceable to the SI via the recently certified IRMM-046b spike reference material in combination with identification and quantification of the sources of uncertainties for IDMS according to the ISO/BIPM Guide to the Expression of Uncertainty in Measurement (GUM) [1].

Figure 3: The mean amount content of ^{239}Pu in IRMM-1027n compared with the measured single values by IDMS (with expanded uncertainties coverage factor *k*=2).



Verification of U amount content in selected vials

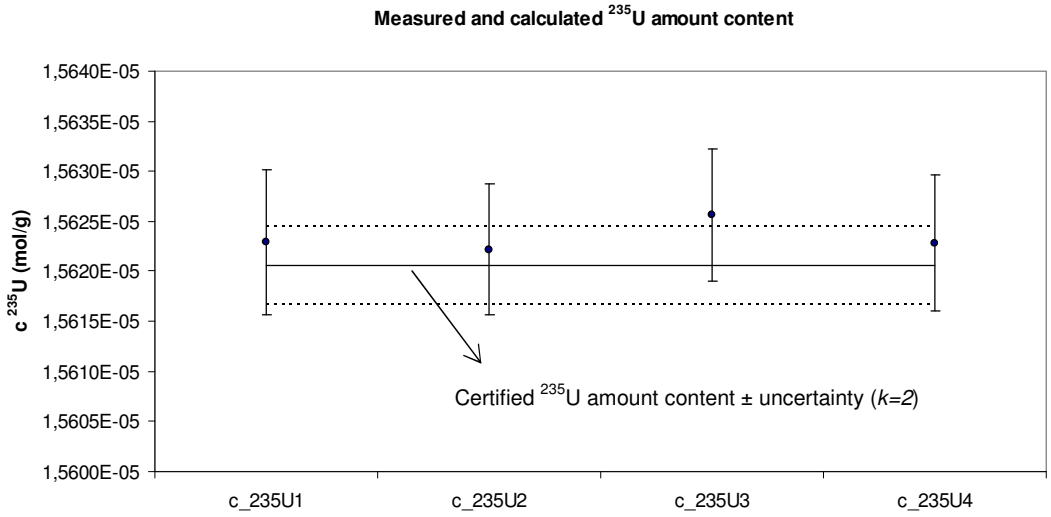
As for plutonium, a set of four results from the measurements used to demonstrate homogeneity was chosen for the verification of the uranium content.

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

Table 3: Amount content of ^{235}U in mol·g^{-1}. Values from metrological weighing are compared with mean values calculated from measurement of individual vials by IDMS. Expanded uncertainties are given in brackets (coverage factor *k*=2).

Certificate	Vials series 046b
1.562 06(39) · 10^{-5}	1.562 34(44) · 10^{-5}

Figure 4: ''Metrological' amount content of ^{235}U in IRMM-1027n (from the masses of metals and solution) compared with the measured values by IDMS (with expanded uncertainties coverage factor *k*=2).



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.

Due to incomplete dissolution of the plutonium reference metal the certification of 1027n is based on mass metrology of the uranium metals and the solutions, the certificate of the base materials, and on IDMS certification measurements for plutonium. The successful verification of the certified values for uranium from mass metrology was accomplished by IDMS measurements on individual vials. For the certification of the plutonium amount content with IDMS a recently certified IRMM-046b spike reference material was used. The materials prepared are available from IRMM, Geel as reference material IRMM-1027n for measurements of uranium and plutonium in fissile material accountancy and control.

References

[1] R. Jakopič, A. Verbruggen, R. Eykens, F. Kehoe, H. Kühn, Y. Kushigeta, U. Jacobsson, J. Bauwens, S. Richter, R. Wellum, Y. Aregbe, An inter-calibration campaign using various selected Pu spike isotopic reference materials, J. Radioanal. Nucl. Chem. (2010), 286, 449-454].

[2] http://www-cetama.cea.fr

[3] 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

[4] 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

[5] An automated system for the preparation of Large Sized Dried (LSD) Spikes, A. Verbruggen, J. Bauwens, N. Van De Steene, U. Jakobsson, R. Eykens, R. Wellum, Y. Aregbe, ATALANTE Conference 2008, Montpellier (France), May 19-22, 2008

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.

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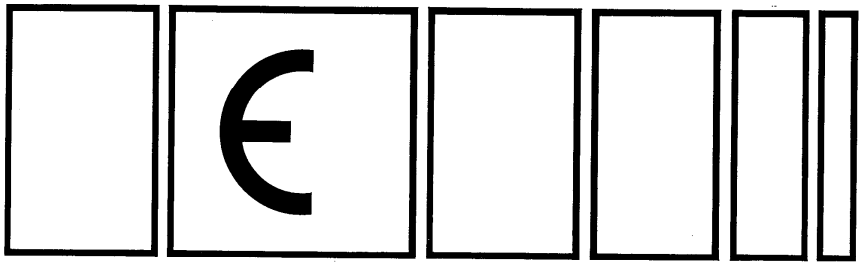

Annex 2: Certificate of uranium metal: NBL CRM-116

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
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

CETAMA certifié ISO 9001:2000 par BVQI

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

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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

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

E. 3688 **Issued date**: 08 June 2010 **Page** 1 **of** 1

Applicant: Verbruggen **Group:** RM-Nuclear

Project: IRMM-1027 N LSD **IM-unit ref.:**

Description: Preparation mother solution IRMM-1027 N

Date of receipt of request: 10 July 2008 **Weighing date**: 19 June 2009

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

	Weight in g
Mass of Pu metal (MP 2 BC 2701)	2.1562 (3)
Mass of U metal (NBL-CRM-116)	11.923 (2)
Mass of U metal (EC-NRM-101)	47.701 (6)
Mass of IRMM-1027 N	3115.26 (7)

Observations:

The measurements and uncertainty estimates, were performed according to working instruction WI-0185, "Mass determination by substitution weighing"on balances AT 261 and At 201 with IRMM inventory No 1999 00337 27 and 1996 00547 73 and balance PR 5002 with reference No9800298.

Traceability:

The certified mass values are traceable to the International Kilogram Prototype via regular calibrations of the IRMM principal kilogram. The sets of working mass standards M 3 and M 10 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: Certificate of IRMM-1027n

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for Reference Materials and Measurements
Reference Materials Unit

CERTIFICATE
SPIKE ISOTOPIC REFERENCE MATERIAL IRMM-1027n

This certified Spike Isotopic Reference Material consists of uranium and plutonium isotopes. The material has been prepared in the following way.

A solution with certified isotope amount contents of

1.562 06(39) 10^{-5} mol (^{235}U) · g^{-1} (solution)
6.473 2(10) 10^{-5} mol (^{238}U) · g^{-1} (solution)
2.791 3(12) 10^{-6} mol (^{239}Pu) · g^{-1} (solution)

was dispensed in penicillin vials, subsequently evaporated to dryness and covered with a dry layer of circa 50 mg cellulose acetate butyrate (CAB) to ensure spike integrity.

Each unit is identified by a vial number. The sample mass of the solution dispensed in each vial is listed in the certificate of weighing.

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

$n(^{234}U)/n(^{238}U)$:	0.002 556(10)
$n(^{235}U)/n(^{238}U)$:	0.241 312(72)
$n(^{236}U)/n(^{238}U)$:	0.001 051 5(32)

$n(^{238}Pu)/n(^{239}Pu)$:	0.000 029 98(28)
$n(^{240}Pu)/n(^{239}Pu)$:	0.022 426 3(51)
$n(^{241}Pu)/n(^{239}Pu)$:	0.000 200 2(26)
$n(^{242}Pu)/n(^{239}Pu)$:	0.000 075 71(78)

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

	amount fraction (·100)		mass fraction (·100)
$n(^{234}U)/n(U)$	0.205 35(83)	$m(^{234}U)/m(U)$	0.202 39(82)
$n(^{235}U)/n(U)$	19.383 7(47)	$m(^{235}U)/m(U)$	19.186 7(47)
$n(^{236}U)/n(U)$	0.084 46(26)	$m(^{236}U)/m(U)$	0.083 96(26)
$n(^{238}U)/n(U)$	80.326 5(44)	$m(^{238}U)/m(U)$	80.527 0(44)

	amount fraction (·100)		mass fraction (·100)
$n(^{238}Pu)/n(Pu)$	0.002 931(28)	$m(^{238}Pu)/m(Pu)$	0.002 919(27)
$n(^{239}Pu)/n(Pu)$	97.773 1(56)	$m(^{239}Pu)/m(Pu)$	97.768 09(56)
$n(^{240}Pu)/n(Pu)$	2.192 78(49)	$m(^{240}Pu)/m(Pu)$	2.201 76(49)
$n(^{241}Pu)/n(Pu)$	0.019 57(26)	$m(^{241}Pu)/m(Pu)$	0.019 74(26)
$n(^{242}Pu)/n(Pu)$	0.007 402(76)	$m(^{242}Pu)/m(Pu)$	0.007 495(77)

The molar mass of the uranium in this sample is 237.458 02(13) $g \cdot mol^{-1}$
The molar mass of the plutonium in this sample is 239.074 790 8(90) $g \cdot mol^{-1}$

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

$8.058\ 6(11) \cdot 10^{-5}$	mol (U) · g^{-1} (solution)
$3.671\ 52(91) \cdot 10^{-3}$	g (^{235}U) · g^{-1} (solution)
$15.409\ 5(24) \cdot 10^{-3}$	g (^{238}U) · g^{-1} (solution)
$19.135\ 8(25) \cdot 10^{-3}$	g (U) · g^{-1} (solution)
$2.854\ 8(12) \cdot 10^{-6}$	mol (Pu) · g^{-1} (solution)
$6.672\ 7(29) \cdot 10^{-4}$	g (^{239}Pu) · g^{-1} (solution)
$6.825\ 0(29) \cdot 10^{-4}$	g (Pu) · g^{-1} (solution)

NOTES

1. The certified values of this Spike Isotopic Reference Material are metrologically traceable to the SI. Measurements calibrated with this Isotopic Reference Material can therefore provide SI-traceable results.

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. IRMM-1027n was prepared by metrological weighing of U metals (NBL CRM 116, EC NRM 101) and Pu metal (CETAMA MP2), dissolving in HNO_3, subsequently dispensing by metrological weighing into individual units, drying and conditioning in cellulose acetate butyrate (CAB). The plutonium amount content was certified by IDMS using the CRM IRMM-046b spike.

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

5. Values for isotope amount ratios, isotopic compositions and concentrations are valid for 01 November 2010. This certificate is valid until November 2012; the validity may be extended after further tests on the stability of the spike material are carried out.

6. It is recommended to store the vials in vertical position.

7. The half lives used in the calculations are

^{238}Pu : 8.77 (03) · 10^1 a[1]
^{239}Pu : 2.411 (03) · 10^4 a[1]
^{240}Pu : 6.563 (07) · 10^3 a[1]
^{241}Pu : 1.432 5(24) · 10^1 a[2]
^{242}Pu : 3.735 (11) · 10^5 a[1]
^{244}Pu : 8.00 (09) · 10^7 a[1]

8. The atomic masses, used in the calculations, are[3]

^{233}U : 233.039 635 2 (58) g·mol^{-1}
^{234}U : 234.040 952 1 (40) g·mol^{-1}
^{235}U : 235.043 929 9 (40) g·mol^{-1}
^{236}U : 236.045 568 0 (40) g·mol^{-1}
^{238}U : 238.050 788 2 (40) 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}

9. 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.

H. Emons
Head Reference Materials Unit

Geel, December 2010

(1) IAEA, Decay data of the Transactinium Nuclides, Technical Reports Series No. 261, 1986
(2) R. Wellum, A. Verbruggen, R. Kessel, J. Anal. At. Spectrom., 2009, 24, 801 - 807
(3) G. Audi and A.H. Wapstra, The 2003 atomic mass evaluation, Nucl Phys A729 (2003) 337-676.

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

E. 3743 **Issue date:** 2010-02-24 **Page** 1 **of** 7

Applicant: Verbruggen **Group:** Nuclear SG **Project:** Lotus Notes ID: 285

Description: 1027N individual vials Nr 1027N-0001 to 1027N-1238

Date of receipt of request: N/A **Weighing date:** 2009-06-22 – 06-26

Results:

The reported results applies only to the objects / samples described in this certificate and are shown in the annex

Observations:

The measurements and uncertainty estimates, were performed according to working instruction RM-WI-0368, "LSD automated system equipment manual" on balance Sartorius TE124 installed in a dispensing robot, Nucomat with IRMM inventory no 2006 00290 17.

Traceability:

The certified mass values are traceable to the International Kilogram Prototype via regular calibrations of the IRMM principal mass standards. The mass standard identified as H208 (cylinder + vial certificate IRMM E3612) was used as verification of balance performance 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 has a value of 0.0006 g for the annexed results.

Annex 1: Weighing results

.....J. Bowen.....
Signature
Nuclear SG analyst

.....
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

Vial No	Mass / g	Vial No	Mass / g	Vial No	Mass / g	Vial No	Mass / g
0001	2.5152	0055	2.5208	0109	2.5111	0172	2.5112
0002	2.5264	0056	2.5260	0110	2.5215	0173	2.5153
0003	2.5211	0057	2.5169	0111	2.5161	0174	2.5184
0004	2.5160	0058	2.5195	0112	2.5251	0175	2.5303
0005	2.5192	0059	2.5312	0113	2.5233	0176	2.5154
0006	2.5222	0060	2.5223	0114	2.5253	0177	2.5207
0007	2.5247	0061	2.5126	0115	2.5169	0178	2.5225
0008	2.5173	0062	2.5260	0116	2.5110	0179	2.5129
0009	2.5250	0063	2.5186	0117	2.5247	0180	2.5228
0010	2.5150	0064	2.5258	0118	2.5192	0181	2.5318
0011	2.5242	0065	2.5298	0119	2.5248	0182	2.5054
0012	2.5174	0066	2.5138	0120	2.5213	0183	2.5262
0013	2.5227	0067	2.5259	0121	2.5175	0184	2.5198
0014	2.5205	0068	2.5226	0122	2.5204	0185	2.5173
0015	2.5163	0069	2.5162	0124	2.5221	0186	2.5182
0016	2.5234	0070	2.5324	0130	2.5260	0187	2.5211
0017	2.5156	0071	2.5118	0134	2.5211	0188	2.5254
0018	2.5242	0072	2.5213	0135	2.5184	0189	2.5302
0019	2.5276	0073	2.5231	0136	2.5218	0190	2.5166
0020	2.5149	0074	2.5321	0137	2.5170	0191	2.5085
0021	2.5199	0075	2.5124	0138	2.5251	0192	2.5213
0022	2.5245	0076	2.5225	0139	2.5212	0193	2.5235
0023	2.5268	0077	2.5225	0140	2.5202	0194	2.5308
0024	2.5159	0078	2.5213	0141	2.5214	0195	2.5090
0025	2.5192	0079	2.5234	0142	2.5217	0196	2.5170
0026	2.5225	0080	2.5224	0143	2.5168	0197	2.5368
0027	2.5176	0081	2.5224	0144	2.5239	0198	2.5061
0028	2.5193	0082	2.5212	0145	2.5220	0199	2.5208
0029	2.5221	0083	2.5232	0146	2.5198	0200	2.5215
0030	2.5235	0084	2.5319	0147	2.5138	0201	2.5173
0031	2.5212	0085	2.5123	0148	2.5227	0202	2.5219
0032	2.5216	0086	2.5215	0149	2.5226	0203	2.5207
0033	2.5210	0087	2.5279	0150	2.5175	0204	2.5210
0034	2.5200	0088	2.5163	0151	2.5213	0205	2.5130
0035	2.5213	0089	2.5278	0152	2.5248	0206	2.5236
0036	2.5232	0090	2.5226	0153	2.5187	0207	2.5232
0037	2.5198	0091	2.5173	0154	2.5215	0208	2.5178
0038	2.5237	0092	2.5222	0155	2.5168	0209	2.5186
0039	2.5214	0093	2.5266	0156	2.5119	0210	2.5255
0040	2.5220	0094	2.5176	0157	2.5323	0211	2.5178
0041	2.5209	0095	2.5257	0158	2.5192	0212	2.5188
0042	2.5197	0096	2.5235	0159	2.5203	0213	2.5201
0043	2.5213	0097	2.5183	0160	2.5242	0214	2.4876
0044	2.5210	0098	2.5197	0161	2.5201	0215	2.5022
0045	2.5250	0099	2.5237	0162	2.5186	0216	2.5221
0046	2.5164	0100	2.5219	0163	2.5205	0217	2.5170
0047	2.5225	0101	2.5221	0164	2.5250	0218	2.5243
0048	2.5274	0102	2.5203	0165	2.5151	0219	2.5174
0049	2.5185	0103	2.5213	0166	2.5237	0220	2.5201
0050	2.5248	0104	2.5214	0167	2.5102	0221	2.5198
0051	2.5200	0105	2.5208	0168	2.5217	0222	2.5219
0052	2.5233	0106	2.5250	0169	2.5342	0223	2.5174
0053	2.5183	0107	2.5216	0170	2.5233	0224	2.5206
0054	2.5234	0108	2.5316	0171	2.5194	0225	2.5201

Vial No	Mass / g	Vial No	Mass / g	Vial No	Mass / g	Vial No	Mass / g
0226	2.5183	0280	2.5099	0334	2.5167	0388	2.5264
0227	2.5222	0281	2.5346	0335	2.5297	0389	2.5109
0228	2.5182	0282	2.5004	0336	2.5055	0390	2.5218
0229	2.5205	0283	2.5199	0337	2.5154	0391	2.5208
0230	2.5193	0284	2.5270	0338	2.5195	0392	2.5103
0231	2.5223	0285	2.5102	0339	2.5177	0393	2.5257
0232	2.5190	0286	2.5339	0340	2.5230	0394	2.5009
0233	2.5167	0287	2.5008	0341	2.5188	0395	2.4920
0234	2.5214	0288	2.5178	0342	2.5226	0396	2.5073
0235	2.5176	0289	2.5178	0343	2.5072	0397	2.5207
0236	2.5203	0290	2.4866	0344	2.5313	0398	2.5159
0237	2.5215	0291	2.5099	0345	2.5065	0399	2.5150
0238	2.5191	0292	2.5042	0346	2.5198	0400	2.5206
0239	2.5197	0293	2.5275	0347	2.5253	0401	2.5180
0240	2.5194	0294	2.5122	0348	2.5095	0402	2.5034
0241	2.5188	0295	2.5195	0349	2.5169	0403	2.5354
0242	2.5190	0296	2.5093	0350	2.5246	0404	2.5205
0243	2.5202	0297	2.5312	0351	2.5110	0405	2.5246
0244	2.5202	0298	2.5137	0352	2.4870	0406	2.4929
0245	2.5214	0299	2.5123	0353	2.5117	0407	2.5449
0246	2.5134	0300	2.5226	0354	2.5058	0408	2.5083
0247	2.5240	0301	2.5107	0355	2.5187	0409	2.5249
0248	2.5159	0302	2.5085	0356	2.5198	0410	2.4917
0249	2.5211	0303	2.5301	0357	2.5192	0411	2.5394
0250	2.5200	0304	2.5040	0358	2.5156	0412	2.5191
0251	2.5187	0305	2.5198	0359	2.5226	0413	2.5160
0252	2.5187	0306	2.5138	0360	2.5196	0414	2.5266
0253	2.5198	0307	2.5172	0361	2.5155	0415	2.5111
0254	2.5138	0308	2.5254	0362	2.5182	0416	2.5164
0255	2.5261	0309	2.5097	0363	2.5179	0417	2.4953
0256	2.5201	0310	2.5242	0364	2.5222	0418	2.5194
0257	2.5187	0311	2.5116	0365	2.5184	0419	2.5342
0258	2.5223	0312	2.5207	0366	2.5215	0420	2.5154
0259	2.5091	0313	2.5129	0367	2.5108	0421	2.5296
0260	2.5339	0314	2.5151	0368	2.5207	0422	2.5154
0261	2.5137	0315	2.5183	0369	2.5202	0423	2.5168
0262	2.5189	0316	2.5182	0370	2.5265	0424	2.4888
0263	2.5215	0317	2.5168	0371	2.5126	0425	2.5156
0264	2.5228	0318	2.5322	0372	2.5159	0426	2.5138
0265	2.5183	0319	2.4999	0373	2.5279	0427	2.5172
0266	2.5117	0320	2.5281	0374	2.5227	0428	2.4906
0267	2.5309	0321	2.5205	0375	2.5070	0429	2.5205
0268	2.5090	0322	2.5044	0376	2.5304	0430	2.5516
0269	2.5207	0323	2.5209	0377	2.5054	0431	2.4958
0270	2.5211	0324	2.5163	0378	2.5233	0432	2.5178
0271	2.5172	0325	2.5169	0379	2.5223	0433	2.5168
0272	2.5073	0326	2.5220	0380	2.5113	0434	2.5096
0273	2.5345	0327	2.5245	0381	2.5283	0435	2.5250
0274	2.5015	0328	2.5049	0382	2.5169	0436	2.5176
0275	2.5337	0329	2.5185	0383	2.5128	0437	2.5152
0276	2.5148	0330	2.5201	0384	2.5270	0438	2.5116
0277	2.5184	0331	2.5151	0385	2.5191	0439	2.5170
0278	2.5089	0332	2.5162	0386	2.5245	0440	2.5153
0279	2.5215	0333	2.5211	0387	2.5118	0441	2.4918

Vial No	Mass / g	Vial No	Mass / g	Vial No	Mass / g	Vial No	Mass / g
0442	2.5392	0496	2.5145	0550	2.5541	0604	2.5031
0443	2.4976	0497	2.5191	0551	2.5143	0605	2.5338
0444	2.5432	0498	2.5415	0552	2.5177	0606	2.5206
0445	2.4891	0499	2.4874	0553	2.5158	0607	2.5053
0446	2.5406	0500	2.5451	0554	2.5013	0608	2.5120
0447	2.5085	0501	2.5519	0555	2.5132	0609	2.5181
0448	2.5117	0502	2.5352	0556	2.5038	0610	2.5110
0449	2.5220	0503	2.5227	0557	2.5123	0611	2.5102
0450	2.5142	0504	2.5188	0558	2.5362	0612	2.5096
0451	2.5145	0505	2.4898	0559	2.4893	0613	2.5054
0452	2.5133	0506	2.5425	0560	2.5298	0614	2.5106
0453	2.5138	0507	2.5124	0561	2.5200	0615	2.5123
0454	2.5129	0508	2.5106	0562	2.5048	0616	2.5525
0455	2.5111	0509	2.4960	0563	2.5486	0617	2.5094
0456	2.5061	0510	2.5443	0564	2.5090	0618	2.5128
0457	2.5169	0511	2.4897	0565	2.5108	0619	2.5257
0458	2.5481	0512	2.5364	0566	2.4912	0620	2.5083
0459	2.5046	0513	2.4934	0567	2.5414	0621	2.5164
0460	2.5329	0514	2.5377	0568	2.5080	0622	2.5527
0461	2.5164	0515	2.5521	0569	2.4903	0623	2.4970
0462	2.5185	0516	2.5110	0570	2.5135	0624	2.5030
0463	2.4919	0517	2.5093	0571	2.5321	0625	2.4975
0464	2.5486	0518	2.5296	0572	2.5114	0626	2.5389
0465	2.5152	0519	2.5010	0573	2.5225	0627	2.5018
0466	2.5537	0520	2.5387	0574	2.5105	0628	2.5189
0467	2.4973	0521	2.5171	0575	2.5138	0629	2.5512
0468	2.4999	0522	2.5210	0576	2.5119	0630	2.4915
0469	2.5182	0523	2.5150	0577	2.5129	0631	2.5068
0470	2.4935	0524	2.5085	0578	2.5005	0632	2.4930
0471	2.5541	0525	2.5147	0579	2.5068	0633	2.5155
0472	2.5542	0526	2.5195	0580	2.5093	0634	2.5261
0473	2.4934	0527	2.5072	0581	2.4967	0635	2.5134
0474	2.5372	0528	2.4918	0582	2.4876	0636	2.5220
0475	2.4959	0529	2.5411	0583	2.5203	0637	2.5081
0476	2.5162	0530	2.5505	0584	2.4929	0638	2.5038
0477	2.5453	0531	2.5017	0585	2.5123	0639	2.4988
0478	2.5528	0532	2.5178	0586	2.5256	0640	2.5229
0479	2.5056	0533	2.5250	0587	2.5415	0641	2.4996
0480	2.5154	0534	2.4925	0588	2.5020	0642	2.5429
0481	2.5230	0535	2.5139	0589	2.5152	0643	2.5139
0482	2.4875	0536	2.5429	0590	2.5174	0644	2.5043
0483	2.5427	0537	2.4896	0591	2.5062	0645	2.4906
0484	2.4941	0538	2.5395	0592	2.5173	0646	2.5311
0485	2.5169	0539	2.5112	0593	2.5204	0647	2.4919
0486	2.5090	0540	2.5130	0594	2.5090	0648	2.5371
0487	2.5440	0541	2.5137	0595	2.4903	0649	2.5490
0488	2.5538	0542	2.5499	0596	2.5294	0650	2.5123
0489	2.5350	0543	2.5291	0597	2.5190	0651	2.5300
0490	2.4928	0544	2.5163	0598	2.4898	0652	2.5108
0491	2.5423	0545	2.5107	0599	2.5314	0653	2.4928
0492	2.5189	0546	2.5133	0600	2.5175	0654	2.5137
0493	2.5166	0547	2.5525	0601	2.5104	0655	2.5225
0494	2.4892	0548	2.5493	0602	2.5511	0656	2.5481
0495	2.5156	0549	2.5153	0603	2.5130	0657	2.5331

Vial No	Mass / g	Vial No	Mass / g	Vial No	Mass / g	Vial No	Mass / g
0658	2.4889	0712	2.5534	0767	2.5174	0821	2.4886
0659	2.5122	0713	2.4894	0768	2.5216	0822	2.5319
0660	2.5440	0714	2.5136	0769	2.5093	0823	2.5108
0661	2.5035	0715	2.5161	0770	2.4910	0824	2.5228
0662	2.5188	0716	2.5055	0771	2.5143	0825	2.5117
0663	2.5077	0717	2.4924	0772	2.5195	0826	2.5118
0664	2.5127	0718	2.5268	0773	2.5067	0827	2.5206
0665	2.5188	0719	2.4919	0774	2.5031	0828	2.5079
0666	2.4889	0720	2.5388	0775	2.5182	0829	2.5057
0667	2.5331	0721	2.4908	0776	2.5519	0830	2.5121
0668	2.5018	0722	2.5461	0777	2.5070	0831	2.5203
0669	2.4947	0723	2.4955	0778	2.5122	0832	2.5151
0670	2.5069	0724	2.5151	0779	2.5093	0833	2.5081
0671	2.5396	0725	2.5165	0780	2.5128	0834	2.5198
0672	2.5091	0726	2.5135	0781	2.4985	0835	2.5110
0673	2.4910	0727	2.5195	0782	2.5164	0836	2.5028
0674	2.5091	0728	2.5519	0783	2.5114	0837	2.4917
0675	2.5027	0729	2.5286	0784	2.5512	0838	2.5170
0676	2.4926	0730	2.5018	0785	2.5091	0839	2.5079
0677	2.5480	0731	2.5080	0786	2.4939	0840	2.4938
0678	2.4894	0732	2.5034	0787	2.5127	0841	2.5008
0679	2.4908	0733	2.5188	0788	2.5072	0842	2.5410
0680	2.5346	0734	2.5526	0789	2.4946	0843	2.4924
0681	2.5221	0735	2.5338	0790	2.5289	0844	2.5428
0682	2.5141	0736	2.5461	0791	2.5127	0845	2.5138
0683	2.5509	0737	2.5067	0792	2.5113	0846	2.5023
0684	2.5167	0738	2.5169	0793	2.5172	0847	2.5164
0685	2.5308	0739	2.5139	0794	2.5051	0848	2.5150
0686	2.5023	0740	2.5040	0795	2.5521	0849	2.5266
0687	2.5103	0741	2.4874	0796	2.5010	0850	2.5154
0688	2.5097	0742	2.5408	0797	2.5189	0851	2.5101
0689	2.5095	0743	2.5385	0798	2.5173	0852	2.5168
0690	2.5199	0744	2.5547	0799	2.5490	0853	2.5143
0691	2.5092	0745	2.4954	0800	2.5306	0854	2.5187
0692	2.4985	0746	2.5116	0801	2.5025	0855	2.5087
0693	2.4953	0747	2.5261	0802	2.5126	0856	2.5187
0694	2.5148	0748	2.5092	0803	2.4874	0857	2.5017
0695	2.5180	0749	2.5169	0804	2.4908	0858	2.5064
0696	2.4968	0750	2.5116	0805	2.5320	0859	2.4993
0697	2.4957	0751	2.5144	0806	2.4958	0860	2.5097
0698	2.5496	0752	2.5152	0807	2.5019	0861	2.5133
0699	2.4953	0753	2.5140	0808	2.5212	0862	2.5434
0700	2.4991	0754	2.5088	0809	2.5181	0863	2.5495
0701	2.5389	0755	2.5081	0810	2.4940	0864	2.5011
0702	2.5091	0756	2.5080	0811	2.5055	0865	2.5129
0703	2.5484	0757	2.4928	0812	2.5153	0866	2.5178
0704	2.4928	0758	2.5212	0813	2.5153	0867	2.5033
0705	2.5484	0759	2.4900	0814	2.5093	0868	2.5195
0706	2.4930	0760	2.5051	0815	2.5063	0869	2.5175
0707	2.5118	0761	2.5362	0816	2.5316	0870	2.5133
0708	2.5183	0762	2.5532	0817	2.4914	0871	2.5159
0709	2.5032	0763	2.4979	0818	2.4906	0872	2.5075
0710	2.5118	0764	2.5220	0819	2.5338	0873	2.5113
0711	2.5205	0766	2.5132	0820	2.5162	0874	2.5532

Vial No	Mass / g	Vial No	Mass / g	Vial No	Mass / g	Vial No	Mass / g
0875	2.5183	0929	2.4918	0983	2.5519	1038	2.5323
0876	2.5102	0930	2.5323	0984	2.5125	1039	2.5506
0877	2.5006	0931	2.5157	0985	2.5545	1040	2.4959
0878	2.5208	0932	2.5475	0986	2.5115	1041	2.5076
0879	2.5495	0933	2.5061	0987	2.5099	1042	2.5124
0880	2.5221	0934	2.5021	0988	2.4901	1043	2.5239
0881	2.4939	0935	2.5259	0989	2.4975	1044	2.5129
0882	2.5093	0936	2.5185	0990	2.5226	1045	2.5490
0883	2.5309	0937	2.5033	0991	2.5140	1046	2.5202
0884	2.5075	0938	2.5159	0992	2.5136	1047	2.4969
0885	2.4921	0939	2.5067	0993	2.4992	1048	2.5300
0886	2.5115	0940	2.5055	0994	2.5101	1049	2.5533
0887	2.5358	0941	2.5010	0995	2.4921	1050	2.4996
0888	2.5057	0942	2.5011	0996	2.5295	1051	2.5480
0889	2.5409	0943	2.5064	0997	2.5380	1052	2.5326
0890	2.5007	0944	2.5189	0998	2.5078	1053	2.5351
0891	2.5036	0945	2.5229	0999	2.4997	1054	2.5132
0892	2.4888	0946	2.5511	1000	2.4952	1055	2.5065
0893	2.5324	0947	2.5000	1001	2.5288	1056	2.5436
0894	2.4920	0948	2.4972	1002	2.4990	1057	2.5060
0895	2.5253	0949	2.5208	1003	2.4987	1058	2.5032
0896	2.4962	0950	2.5072	1004	2.5073	1059	2.5008
0897	2.5366	0951	2.4949	1005	2.5282	1060	2.5182
0898	2.5429	0952	2.5205	1006	2.5147	1061	2.5103
0899	2.4879	0953	2.5433	1007	2.5041	1062	2.5463
0900	2.4911	0954	2.5218	1008	2.5148	1063	2.4964
0901	2.5414	0955	2.5229	1009	2.5427	1064	2.4871
0902	2.5113	0956	2.5198	1010	2.5508	1065	2.5422
0903	2.5518	0957	2.5268	1012	2.4993	1066	2.5057
0904	2.5013	0958	2.5074	1013	2.5251	1067	2.5075
0905	2.5237	0959	2.5107	1014	2.5061	1068	2.5051
0906	2.5026	0960	2.5111	1015	2.5150	1069	2.5471
0907	2.4924	0961	2.5181	1016	2.5205	1070	2.5528
0908	2.5388	0962	2.5016	1017	2.5035	1071	2.4981
0909	2.5516	0963	2.5098	1018	2.5134	1072	2.5209
0910	2.5240	0964	2.5032	1019	2.4949	1073	2.5238
0911	2.5463	0965	2.5184	1020	2.5036	1074	2.4923
0912	2.5015	0966	2.5009	1021	2.5510	1075	2.4946
0913	2.5068	0967	2.5039	1022	2.5316	1076	2.5363
0914	2.5162	0968	2.5149	1023	2.5380	1077	2.5515
0915	2.4912	0969	2.5024	1024	2.5141	1078	2.4908
0916	2.5274	0970	2.5224	1025	2.5078	1079	2.5499
0917	2.4989	0971	2.5039	1026	2.5104	1080	2.4991
0918	2.5267	0972	2.5175	1027	2.5175	1081	2.5105
0919	2.4901	0973	2.5020	1028	2.5097	1082	2.5513
0920	2.5009	0974	2.5510	1029	2.4945	1083	2.5096
0921	2.4915	0975	2.5202	1030	2.5457	1084	2.5475
0922	2.5414	0976	2.5061	1031	2.5378	1085	2.5052
0923	2.5000	0977	2.4983	1032	2.5164	1086	2.5171
0924	2.5039	0978	2.5125	1033	2.5104	1087	2.5441
0925	2.4931	0979	2.5528	1034	2.5058	1088	2.5031
0926	2.5360	0980	2.5106	1035	2.4959	1089	2.5204
0927	2.5527	0981	2.5108	1036	2.5146	1090	2.5006
0928	2.5075	0982	2.5246	1037	2.5028	1091	2.5092

Vial No	Mass / g	Vial No	Mass / g	Vial No	Mass / g	Vial No	Mass / g
1092	2.5232	1129	2.4941	1166	2.5326	1203	2.5007
1093	2.5010	1130	2.5102	1167	2.5476	1204	2.4927
1094	2.5081	1131	2.5317	1168	2.5146	1205	2.5045
1095	2.5131	1132	2.5531	1169	2.5163	1206	2.5389
1096	2.5040	1133	2.5276	1170	2.5468	1207	2.5496
1097	2.5508	1134	2.4960	1171	2.4891	1208	2.4924
1098	2.5037	1135	2.5073	1172	2.5067	1209	2.5069
1099	2.5219	1136	2.4872	1173	2.4959	1210	2.4874
1100	2.5495	1137	2.5374	1174	2.5070	1211	2.5110
1101	2.4908	1138	2.4998	1175	2.5567	1212	2.5341
1102	2.5134	1139	2.4930	1176	2.4970	1213	2.5074
1103	2.4995	1140	2.5315	1177	2.5421	1214	2.5014
1104	2.5491	1141	2.5376	1178	2.4991	1215	2.4923
1105	2.5284	1142	2.5530	1179	2.5482	1216	2.5018
1106	2.5156	1143	2.4891	1180	2.5295	1217	2.5207
1107	2.5211	1144	2.5488	1181	2.5511	1218	2.5075
1108	2.5063	1145	2.4967	1182	2.4903	1219	2.5035
1109	2.4941	1146	2.5225	1183	2.5087	1220	2.4873
1110	2.5237	1147	2.5187	1184	2.5109	1221	2.5073
1111	2.5114	1148	2.5081	1185	2.4949	1222	2.4995
1112	2.5110	1149	2.5091	1186	2.5054	1223	2.4885
1113	2.4950	1150	2.5023	1187	2.5401	1224	2.5239
1114	2.5417	1151	2.5087	1188	2.5447	1225	2.5249
1115	2.4911	1152	2.5415	1189	2.5444	1227	2.5504
1116	2.5044	1153	2.5252	1190	2.4894	1228	2.5190
1117	2.5097	1154	2.5314	1191	2.5164	1226	2.5002
1118	2.5466	1155	2.5086	1192	2.5100	1229	2.5264
1119	2.5029	1156	2.4994	1193	2.5230	1230	2.5050
1120	2.5142	1157	2.5009	1194	2.5530	1231	2.5183
1121	2.5191	1158	2.5162	1195	2.5053	1232	2.5074
1122	2.5004	1159	2.5058	1196	2.5119	1233	2.5055
1123	2.5029	1160	2.5012	1197	2.5060	1234	2.5228
1124	2.5018	1161	2.5160	1198	2.5054	1235	2.4887
1125	2.5496	1162	2.5053	1199	2.4961	1236	2.5302
1126	2.5287	1163	2.5423	1200	2.5356	1237	2.4899
1127	2.5052	1164	2.4978	1201	2.4994	1238	2.5029
1128	2.5218	1165	2.4878	1202	2.5189		

European Commission

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Abstract
Large-sized dried spikes (LSD) have become a fundamental part of the fissile material control of irradiated nuclear fuel. Within the programme at IRMM to provide these spikes to the nuclear industry and the safeguards' inspectorate, a new set of LSD Spikes for the determination of uranium and plutonium by isotope dilution mass spectrometry (IDMS) in solutions of spent fuel from reprocessing plants has been prepared and certified for uranium and plutonium isotopic contents. The methodology followed was comparable to that of previous batches. The solution, made by dissolution of the starting materials in nitric acid, was dispensed directly into individual penicillin vials. An automated system was used to dispense and weigh the vials.
The new batch of large size dried spikes contains ca. 50 mg of uranium with a ^{235}U amount fraction of 19.5% and ca. 1.8 mg of plutonium with a ^{239}Pu amount fraction of 97.8% in each individual vial, covered with a light layer of organic material (cellulose acetate butyrate) as stabilizer.
The U amount content was certified based on values from mass metrology of the validated automated system. Verification of the U amount contents of the spike was done by IDMS at IRMM. The values measured for the dried covered spikes agreed well with those calculated from the masses of starting materials dissolved and the masses of the final solution. The Pu amount content was certified by using IDMS.

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