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Project Report for Certified Reference Material 116-A: Unit Production and Certification of U Amount Content and U Isotope-Amount Ratios

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NBL Program Office
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Summary:

This report describes the production and characterization of a reference material to replace Certified Reference Material 116 (CRM 116). CRM 116 was a highly 235U enriched metal reference material for amount content analysis (assay). Sale of CRM 116 was halted as the metal pieces comprising the CRM developed a planar parting that resulted in inconsistent assay value measurements. Accordingly, it was decided to produce a replacement material designated CRM 116-A.

Each unit of CRM 116-A consists of a 1.1-gram cylinder of metal comprised of ~93% ^{235}U enriched uranium. This report describes the first production run of the CRM 116-A units, the analysis of uranium amount content, and the determination of certified U isotope-amount ratios (isotopic composition). Values and uncertainties from this work (Table I) were incorporated into a certificate of analysis.

Table 1
Certified Attribute Values for CRM 116-A

Amount Content	**Value**	**Uncertainty**	**Isotope-Amount Ratio**	**Value**	**Uncertainty**
g U•g^{-1} metal	0.99945	0.00014	$n(^{233}\text{U})/n(^{235}\text{U})$	0.0000003863	0.0000000086
Molar Mass:	**Value**	**Uncertainty**	$n(^{234}\text{U})/n(^{235}\text{U})$	0.0115836	0.0000097
			$n(^{236}\text{U})/n(^{235}\text{U})$	0.0094713	0.0000077
g•mol^{-1}	235.18572	0.00011	$n(^{238}\text{U})/n(^{235}\text{U})$	0.051277	0.000041
Isotope-Amount Fraction (•100)	**Value**	**Uncertainty**	**Isotope Mass Fraction (•100)**	**Value**	**Uncertainty**
$n(^{233}\text{U})/n(\text{U})$	0.00003603	0.00000080	$m(^{233}\text{U})/m(\text{U})$	0.00003570	0.00000079
$n(^{234}\text{U})/n(\text{U})$	1.08023	0.00089	$m(^{234}\text{U})/m(\text{U})$	1.07497	0.00088
$n(^{235}\text{U})/n(\text{U})$	93.2547	0.0038	$m(^{235}\text{U})/m(\text{U})$	93.1985	0.0038
$n(^{236}\text{U})/n(\text{U})$	0.88324	0.00071	$m(^{236}\text{U})/m(\text{U})$	0.88647	0.00071
$n(^{238}\text{U})/n(\text{U})$	4.7818	0.0036	$m(^{238}\text{U})/m(\text{U})$	4.8401	0.0037

All uncertainties are expanded uncertainties ($U=k\bullet u_c$) with a coverage factor (k) of ≈2 except for the ^{233}U attribute values which have a coverage factor (k) = 3.3 and the amount content value with a coverage factor (k) = 2.3.

Background Information:
In 2008, New Brunswick Laboratory contacted the Y-12 National Security Complex regarding procurement of a homogenous High Enriched Uranium (HEU) metal for a CRM production run. Y-12 had previously performed an extensive study on typical HEU stock material (Jollay, 2007) which indicated that a standard metal “161 casting” (hollow cylinder of U metal, colloquially referred to as a “log”) will yield a relatively homogenous material for most elemental contaminants. A HEU metal log was identified from the Y-12 stock as a candidate material for CRM 116-A (AP3C19-YW-2055) and characterization data for the log was forwarded to NBL. Characterization analyses and unit production were completed in the first half of 2013.

Production of 1.1-gram Metal Samples:
Approximately 500 1.1-g metal pellets (Figure I) were created as the initial production run of Certified Reference Material 116-A. The pellets were produced in a 6 step process:

1. Metal wedges were cut from the designated U metal casting -- AP3C19-YW-2055.
2. The metal wedges were machined to produce 0.5” diameter rods up to 7” long.
3. These rods were then swaged down to approximately 0.25” and annealed in a furnace.
4. The annealed rods were further swaged down to a diameter of 0.17” (changed from 0.125” for initial production) and parted into 4” long pieces.
5. The 0.17” rods were turned on a lathe to remove any irregularities and surface scale.
6. Finally, to produce the 1.1-g individual samples, the machined rods were cut into 0.16” long pieces (changed from 0.3”) on a lathe.

Figure I

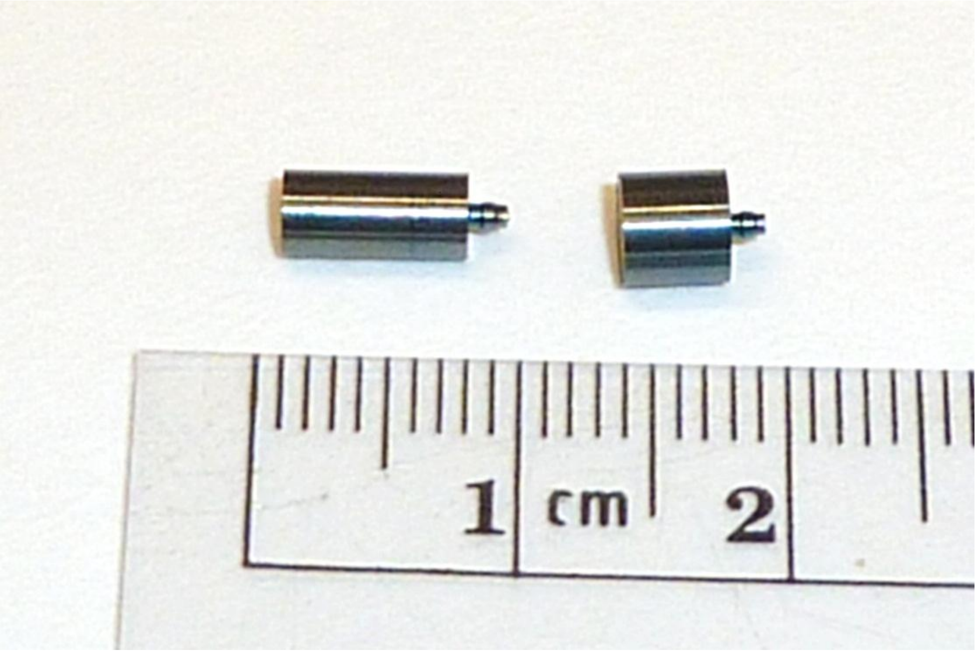


Figure I shows two U metal CRM 116-A pellets. The dimensions of the longer pellet represent the size that was initially planned. The wider pellet is the revised size and is representative of the majority of CRM 116-A units — 470 out of 500. Note, however, that both pellets are ~1.1 g in mass. The nubs on the face of the pellets are remnants from cutting of individual samples using a lathe.

CRM Unit Production:
A packaging work plan was developed specifically for this project. The stock material for unit production was delivered to NBL in five convenience bottles with each containing 40 to 50 grams of metal. The metal pieces were prepared for packaging by placing the contents of each convenience bottle into a beaker and rinsing the samples with ~25 ml of ACS grade acetone to remove any cutting oil. After rinsing, the metal pellets were allowed to dry. Then each pellet was placed into a small zip-top bag which was, in turn, placed into a snap-cap Savillex vial. Each vial was heat-sealed in Mylar and placed into a blue cardboard sample cylinder. Finally, each cylinder was labeled and sealed with shrink sleeving.

Characterization Analyses for Certification:
The overall plan outlines the analyses for determination of U amount content and the U isotope amount ratios for CRM 116-A. The primary amount content determination method was the NBL High-Precision Titrimetric method (HPT), with NBL Titrimetric method (Davies & Gray: D&G) and Isotope Dilution Mass Spectrometry (IDMS) performed as confirmatory analyses and to evaluate sample-to-sample homogeneity. Isotope amount ratios were measured on three mass spectrometers (U MAT 261, U Triton, and Pu Triton) using three analysis techniques. The distribution plan for the characterization of samples provided to NBL was designed so that each analysis technique would characterize samples from throughout the production run.

Uranium Amount Content Determination:
The initial characterization analyses performed by Y-12, indicate that the HEU metal is greater than 99.94% uranium. For this project, the U amount content was determined and verified using the HPT method, the D&G method, and IDMS. The National Institute of Standards and Technology (NIST) SRM 136f Potassium Dichromate Oxidimetric Standard was used for the D&G titrimetric method, New Brunswick Laboratory's CRM 99 was used for the HPT measurements, and CRM 112-A was used as a tracer for IDMS. The D&G and IDMS analyses were performed to identify any sample heterogeneity, while HPT titrations on 1.1-g samples were performed to quantify any potential variability between individual sample units and 3-g HPT analysis was conducted to determine a precise overall assay value.

Uranium Amount Content Results:
The certified amount content value with expanded uncertainty is shown in Table I.

The D&G and 1.1-g HPT measurements appear to indicate that the samples are heterogeneous at the 1.1 gram unit size. An Analysis of Variance (ANOVA) for the D&G data sets yields an "F statistic" of 5.25 which is higher than the "F-critical" value of 3.11, indicating that at the 99% confidence level there is significant sample-to-sample variability.

The IDMS data appears to be biased relative to the HPT and D&G data, and the distribution of values might also be indicative of the sample variation. The repeatability and uncertainty of the IDMS measurements, however, are not sufficient to distinguish sample-to-sample differences at this level. It is also noteworthy that the three samples analyzed by both methods do not indicate a parallel distribution pattern; i.e. the samples with low relative amount content values by D&G analyses do not correspond to low relative amount content values for IDMS measurements.

Figure II shows the mean value of the measured 3-g HPT, 1.1-g HPT, D&G, and IDMS analysis for U amount content in the CRM 116-A material, as well as the mean value of determinations made at Y-12. Error bars are the standard deviation for each data set or standard uncertainty for HPT and IDMS. The proposed certificate value and the limits of the expanded uncertainty for C116-A are also shown.

Figure II

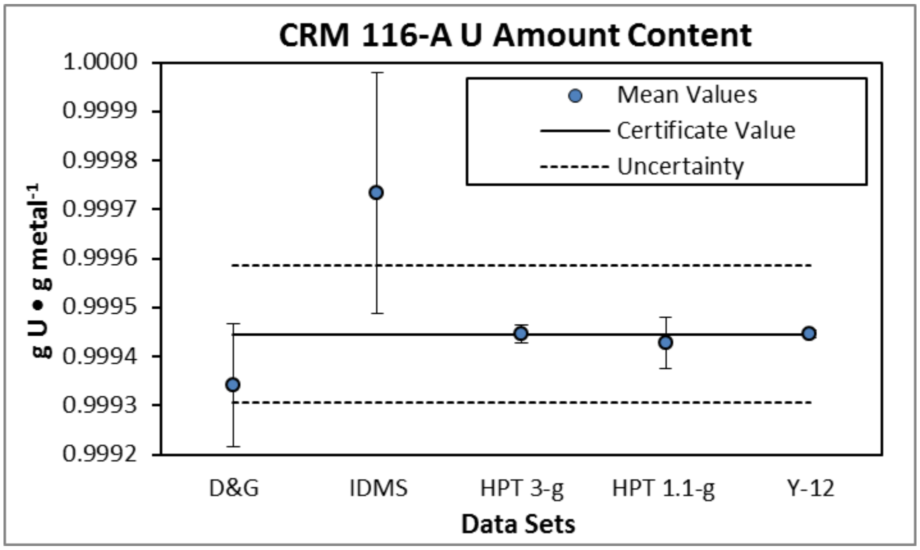


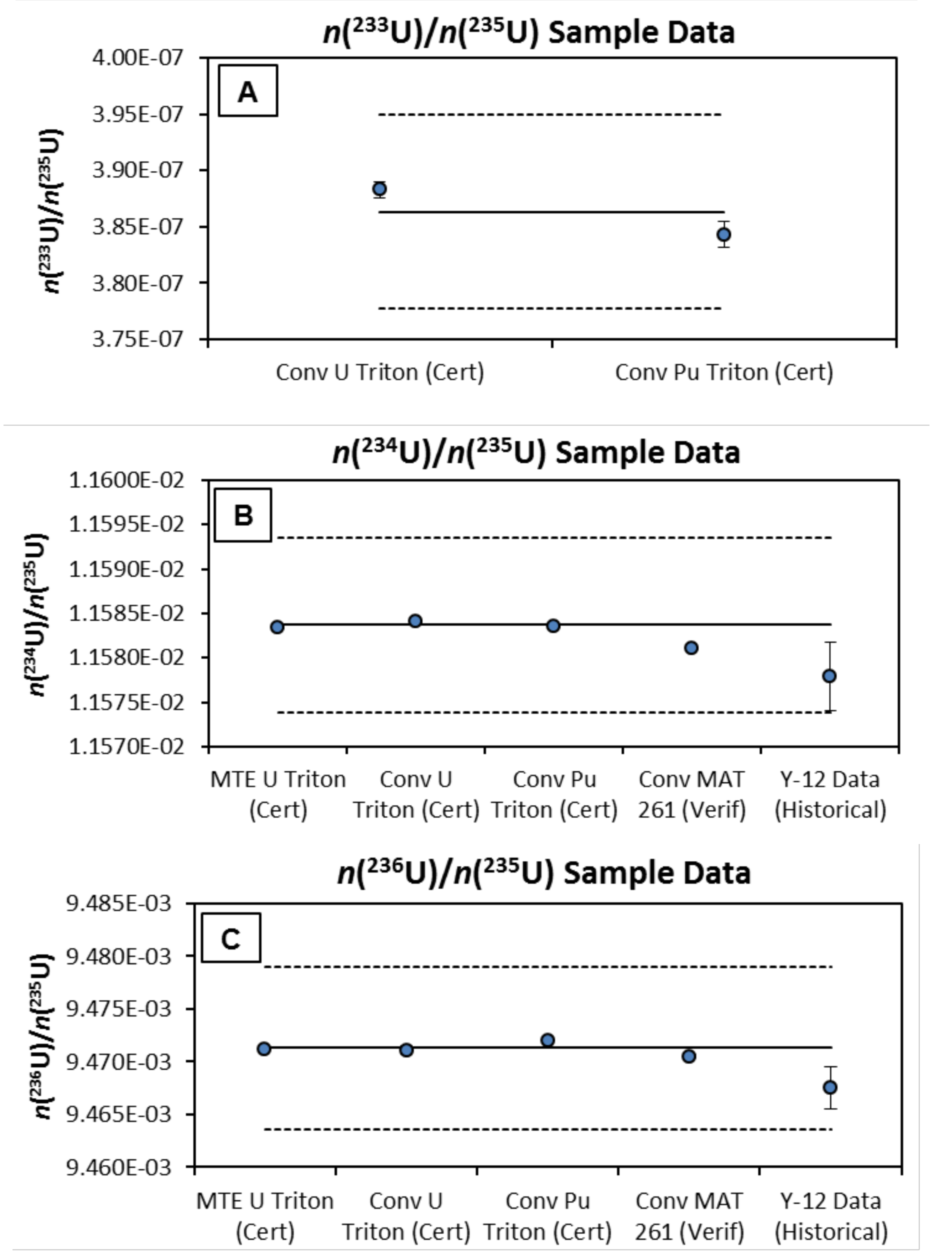
Figure II shows assay data by method.
Error bars for Y-12 data are smaller than the symbol.

Uranium Isotope Amount Ratio Determination:
Three different analysis methods were used for isotopic ratio measurements. These methods were 1) "classical" Total Evaporation (TE: Callis and Cappis, 1994); 2) Modified Total Evaporation (MTE: Richter and Goldberg, 2004); and 3) conventional analysis with internal normalization and the 233U isotope measured and a Secondary Electron Multiplier (SEM). CRMs U900 and U930-D were used for instrumental mass bias calibration and U970 was measured for quality control.

Uranium Isotope-Amount Ratio Results:
The isotope-amount ratios and associated uncertainties shown in Table I were determined from 58 separate n(^{238}U)/n(^{235}U) values, 32 separate n(^{233}U)/n(^{235}U) values and 68 analyses of n(^{234}U)/n(^{235}U) and n(^{236}U)/n(^{235}U). For verification purposes, data are provided from a single TE turret analyzed for the n(^{235}U)/n(^{238}U) ratio on the Pu Triton and from two turrets of conventional analyses for the n(^{234}U)/n(^{238}U) and n(^{236}U)/n(^{238}U) ratios measured on the MAT 261. Combined data for each analysis method/instrument, along with the certified values and expanded uncertainties, are shown in Figures III A through III D. This figure includes verification data and characterization data from Y-12. Note that the error bars for each measurement method-instrument combination are standard uncertainties.

No statistically significant sample-to-sample variability is observed for any of the isotope-amount ratios. Statistically significant turret-to-turret differences were, however, observed for some isotopic ratios within methods and for all ratios between methods (except for n(^{233}U)/n(^{235}U) which was only analyzed by a single method on two instruments). It should be noted that these statistically significant differences are only recognizable because of the relatively tight measurement precision (all %RSD <0.015%) and large data sets (≥ 29 measured values per ratio-method). For instance the relative difference of the mean values for the different methods for n(^{234}U)/n(^{235}U), n(^{236}U)/n(^{235}U) ,and n(^{238}U)/n(^{235}U) are 0.003%, 0.007% and 0.03% respectively.

Figure III (A-D)



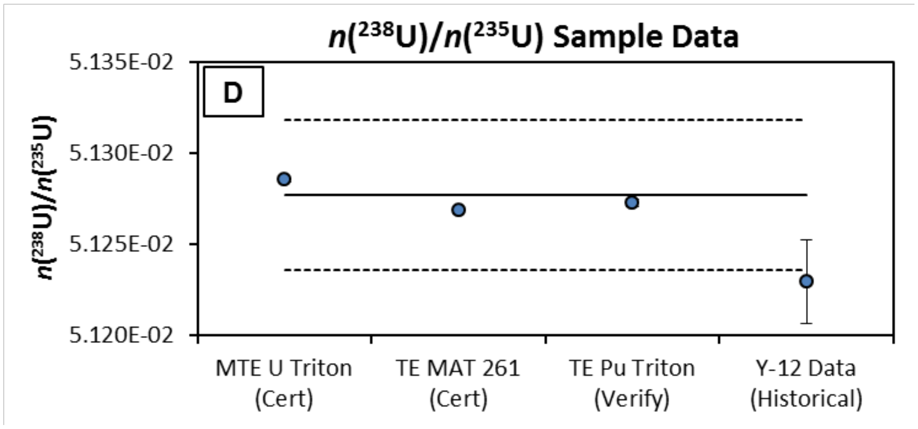


Figure III (A-D) shows the distribution of measured isotopic ratios by analysis method. Error bars are standard uncertainties and are frequently smaller than the chart symbols.

Discussion:

The project described in this report incorporates three major efforts: 1) creation and packaging of 1.1 gram samples of enriched uranium metal for use as an amount content and isotope-amount ratio standard - CRM 116-A; 2) determination of a GUM-compliant certified value and uncertainty for uranium amount content; and 3) determination of GUM-compliant certified values and uncertainties for uranium isotope-amount ratios.

A CRM is defined as *“material characterized by a metrologically valid procedure for one or more specified properties, accompanied by a certificate that provides the value of the specified property, its associated uncertainty, and a statement of metrological traceability”* (ISO 2008). Detailed requirements for production of a certified reference material are outlined in ISO Guide 34 and ISO Guide 35. These requirements include evaluation of material stability and homogeneity, metrological traceability, reproducibility, and the assignment of GUM compliant measurement uncertainties. The certification project for the CRM 116-A was planned and executed specifically to meet these requirements and provide the documentation necessary to certify amount content and isotope-amount ratios.

Stability:

A CRM 116-A unit consists of a small cylinder of U metal. U metal is not stable under atmospheric conditions, readily oxidizing, and all U metal CRMs form an oxide surface coating while the bulk of the metal material remains intact underneath. The metal was packaged by NBL in a manner that will minimize exposure to oxidizing conditions, prevent any oxide coating from being dispersed during storage, and also prevent any rattling that might lead to enhanced degradation of the sample due to mechanical processes. The certificates for these materials include specific instructions for removing the oxide for quantitative analytical use of the metal and, under normal storage conditions, CRM 116-A should last indefinitely.

Homogeneity:

To use a CRM 116-A sample as an analytical reference material requires the complete dissolution of a single unit. The CRM’s attribute values and uncertainties are based on the smallest unit size, nominally 1.1 g. The HPT and D&G titrations performed on 1.1-g samples appear to show statistically significant sample-to-sample differences. These differences are not reflected by the IDMS data nor was any systematic variability across the production run evident from the larger (~3 g) HPT analyses. Furthermore, the HPT data is totally consistent with the characterization data provided by Y-12 which covers a broader distribution of sampling locations on the stock HEU log. To account for the potential variability the standard deviation (as opposed to the standard uncertainty) observed for the 1.1-g HPT samples was used in the calculation of the uncertainty for the CRM 116-A U amount content.

The isotope-amount ratio data, for the eight samples analyzed, do not show statistically significant sample-to-sample heterogeneity. Although small but statistically significant method-to-method and turret-to-turret differences were observed, sample heterogeneity was not identified in either method-specific or combined data sets, despite the small relative variability of most of the isotopic ratio measurements (%RSD <0.02%).

Traceability:
To establish the traceability of the certified attributes for CRM 116-A it is necessary to demonstrate that the values *"can be related, with a stated uncertainty, to stated references, usually national or international standards, thorough an unbroken chain of comparisons"* (ISO 1992). The attributes certified for this material are the U amount content and the isotope amount ratios. These attributes have separate traceability chains but both are tied directly to the national metrology data base for the United States as maintained by the National Institute of Standards and Technology (NIST- formerly National Bureau of Standards).

Reproducibility:
The general accuracy and reproducibility of measured attributes can be assessed by comparing the results of independent measurements. The amount content values for the twelve 3-g samples analyzed by High Precision Titration are, within uncertainties, indistinguishable from the 1.1-g HPT and D&G values and are essentially identical to the values obtained by Y-12 for the starting material.

There is no readily available method for determination of U isotope-amount ratios that is sufficiently precise to serve as an independent confirmation method for magnetic sector mass spectrometry. Therefore, to assure reproducibility of results, multiple mass spectrometry measurement methods and measurement instruments were used to determine certified values and to perform verification measurements (Table II). The magnitude of any recognized instrument or method biases were quantified and incorporated into the measurement uncertainties (See Measurement Uncertainty section, below).

The independent Y-12 measurements and the verification measurements performed for this study on the n(^{234}U)/n(^{235}U), n(^{236}U)/n(^{235}U) ,and n(^{238}U)/n(^{235}U) isotope amount ratios are all consistent with the certified values and uncertainties. Independent characterization data are not available for the n(^{233}U)/n(^{235}U) isotope amount ratio. However, two separate instruments were used to make the measurements and the uncertainties are sufficiently expansive to encompass reasonably expected variability.

Table II
Independent Amount Content and Isotope-Amount Ratio Determinations

Determination	**Amount Content (g U • g Metal^{-1})**	**Standard Uncertainty**
3-g HPT	0.999445	0.000005
1.1-g HPT	0.999427	0.000021
D&G Titration	0.999342	0.000051
IDMS	0.999733	0.000071
U by Diff (Y-12)	0.999445	0.000004
Certified Value[1]	**0.99945**	**0.00014**

Determination	**$n(^{234}\text{U})/n(^{235}\text{U})$**	**Standard Uncertainty**	**$n(^{236}\text{U})/n(^{235}\text{U})$**	**Standard Uncertainty**	**$n(^{238}\text{U})/n(^{235}\text{U})$**	**Standard Uncertainty**
Y-12 Isotopic Data	0.0115779	0.0000038	0.0094675	0.0000020	0.051229	0.000023
Verification Mass Spec[2]	0.0115811	0.0000002	0.0094705	0.0000001	0.0512727	0.0000021
Certified Values[1]	**0.0115836**	**0.0000097**	**0.0094713**	**0.0000077**	**0.051277**	**0.000041**

[1] Uncertainties for the certified value are expanded uncertainties (see Table 1). [2] Verification mass spectrometry analyses include conventional analysis with internal normalization for minor ratios on the MAT 261, and Total Evaporation on the Pu Triton for the $n(^{238}\text{U})/n(^{235}\text{U})$ ratio.

Measurement Uncertainty:
Uncertainties of the measurement results were calculated in accordance with the JCGM 100:2008 Guide for the Expression of Uncertainty in Measurement (GUM) using the Workbench software developed by Metrodata (2009) in combination with “R” open source statistical program. The measurement uncertainties include all recognized significant Type-A and Type-B evaluated sources of uncertainty.

The uncertainty estimated for U amount content included two Type A components, the variability of the 3-g HPT measurements expressed as the standard uncertainty for 12 measurements and the variability of the 1.1-g HPT measurements expressed as the standard deviation of six measurements. The standard deviation of the 1.1-g sample data was used in the uncertainty calculation to account for low-level heterogeneity that could be present in the HEU material. The Type B component for the amount content is the uncertainty for the oxidimetric potential of the CRM 99 material used for the HPT titrations. No component for weighing uncertainty was included in the evaluation because the random component of a weighing uncertainty will be confounded in the variability of multiple measurements and the potential bias would contribute 1% or less to the total uncertainty.

For the $^{238}\text{U}/^{235}\text{U}$ isotope ratio, the following sources of Type-A evaluated uncertainty were included: variability in the major isotope ratio as represented by replicate measurements of the sample; variability in the mass fractionation correction factor within sample turrets as represented by replicate comparator measurements on each turret; and turret-to-turret and method-to-method variability components calculated to account for a previously described variability between MTE turrets and between MTE and TE methods. Type-B evaluated uncertainty components included uncertainty for the $^{235}U/^{238}U$ ratios determined from the certified values of the comparator CRMs.

For estimation of the uncertainties on the $^{233}U/^{235}U$, $^{234}U/^{235}U$, and $^{236}U/^{235}U$ ratios, several components were included, such as the Type-A variability in the minor isotope ratio measurements as represented by replicate measurements of the sample and turret-to-turret and method-to-method variability components, as appropriate. Type-B evaluated uncertainties include those associated with background corrections at ^{233}U, ^{234}U, and ^{236}U. For the $^{233}U/^{235}U$, $^{234}U/^{235}U$, and $^{236}U/^{235}U$ ratio background corrections, conservative estimates of the uncertainty factors, equivalent to half of the mean value between the high and low mass backgrounds, were applied to the data. The $^{233}U/^{235}U$ ratio included additional Type-B evaluated components for uncertainty associated with SEM-Faraday gain calibrations and an interference at atomic mass 233 that has been identified on blank filaments (probably $^{185}Re^{16}O_3$). For all three minor ratios, a Type-B evaluated uncertainty component was also included for internal normalization (i.e. mass fractionation correction). This uncertainty component corresponds to the relative combined standard uncertainty of the measured CRM 116-A $^{238}U/^{235}U$ ratio used for the internal normalization of the minor isotope ratios.

Several potential uncertainty components were not explicitly included in the models for the isotope-amount ratios. The variability associated with amplifier baseline and gain calibrations are not separable from the observed variability of the replicate measurements, so to include components for these potential uncertainty sources would essentially be double counting. Additionally, the MassCom SEMs used for the analyses (model number: MC-TE-Z17 Triton) appears to be highly linear over the range signal intensities relevant to this project (1,000 cps for ^{233}U to 300,000 cps for cup efficiency determination) and the darknoise for the SEMs was observed to be substantially less than 0.1 cps. Accordingly, the potential contributions to the total uncertainty are so small (<1% contribution) that no uncertainty components were included for the SEM linearity or dark noise. Finally, Faraday cup efficiency is a potential source of bias in multi-collector instruments but this effect is difficult to isolate. Cup efficiency effects for the $n(^{238}U)/n(^{235}U)$ ratios (if any) are confounded with the mass bias correction, so no correction is appropriate. For minor ratios, the effect is assumed to be negligible relative to other sources of bias and uncertainty such as the mass bias corrections and the background corrections. This assumption is consistent with the lack of significant instrument-to-instrument bias and the small relative magnitude of the method-to-method bias for minor ratio measurements despite different cup configurations.

The uncertainty for the $^{234}U/^{235}U$, $^{236}U/^{235}U$, and $^{238}U/^{235}U$ isotopic ratios for CRM 116-A are dominated by the uncertainty for the $^{235}U/^{238}U$ ratio derived from the certified U abundances in the CRM U900 and U930-D comparators (combined relative expanded uncertainty of 0.065%), which were used to correct for mass fractionation in the MTE and TE experiments. Uncertainty for the $^{233}U/^{235}U$ ratio is dominated by the uncertainty on the interference correction, the turret-to-turret variability, and the SEM-Faraday gain calibration, while the measurement variability and the background correction contributed to a lesser extent.

Interlaboratory Comparison:

JRC-Geel performed isotopic comparison measurements by Thermal Ionization Mass Spectrometry (TIMS) of NBL Certified Reference Materials C115 DU metal in 2014 and C116-A HEU metal in 2015. These measurements were performed under the auspices of the cooperative safeguards agreement between DOE and EURATOM, under Action Sheet 43 (AS43).

The objective of Action Sheet 43 is to strengthen the traceability of accountability measurements and ensure adequate quality of reference materials and safeguards laboratory measurements through coordination between JRC-Geel and NBL, being two primary providers of nuclear reference materials. JRC-Geel and NBL are not only providing certified nuclear reference materials (CRMs) but also have a long history as interlaboratory comparison providers to most of the world's nuclear facilities, governments, safeguards organizations and laboratories in the field. AS43 links NBL and JRC-Geel's measurement systems to ensure standard and traceable measurement quality at the highest metrological level, supporting international inspectorates with the capability to conduct independent measurements to verify operator declarations that are of high quality and traceable to the SI. Specific tasks in AS43 are the mutual participation in JRC-Geel REIMEP/NUSIMEP and SME (Standard Measurement Evaluation) interlaboratory comparisons, the availability of high-priority reference material and providing verification measurements for CRMs.

These uranium metal CRM's are used widely for isotopic and uranium content, measurement calibration, quality control, measurement validation, and in the case of CRM 116-A serve as a base material for production of spikes used by analytical labs at reprocessing facilities for accountancy. The JRC-Geel results agreed well with the NBL certified property values. The results are in the report: Interlaboratory Comparison of New Brunswick Laboratory (NBL) Depleted Uranium Metal CRM 115 and Highly Enriched Uranium Metal CRM 116-A; M. Soriano, P. Croatto, P. Mason (US Department of Energy NBL Program Office); S. Richter (European Commission – Joint Research Center-Geel (EC-JRC-Geel)).

Conclusion:

Proposed certified values and uncertainties for CRM 116-A are presented in Table I. Traceable attribute values with GUM compliant uncertainties are provide for the U amount content, the n(^{233}U)/n(^{235}U), n(^{234}U)/n(^{235}U), n(^{236}U)/n(^{235}U), and n(^{238}U)/n(^{235}U) isotope amount ratios, and the isotope amount fraction and isotope mass fractions derived from the isotope amount ratios. Accordingly, the HEU metal characterized in this study is suitable for distribution as Certified Reference Material 116-A.

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