

## Standard Test Method for Measurement of <sup>235</sup>U Fraction Using Enrichment Meter Principle<sup>1</sup>

This standard is issued under the fixed designation C 1514; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method covers the quantitative determination of the fraction of  $^{235}$ U in uranium using measurement of the 185.7 keV gamma-ray produced during the decay of  $^{235}$ U.

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1.2 This test method is applicable to items containing homogeneous uranium-bearing materials of known chemical composition in which the compound is considered infinitely thick with respect to 185.7 keV gamma-rays.

1.3 This test method can be used for the entire range of  $^{235}$ U fraction as a weight percent, from depleted (0.2 %  $^{235}$ U) to highly enriched (97.5 %  $^{235}$ U).

1.4 Measurement of items that have not reached secular equilibrium between <sup>238</sup>U and <sup>234</sup>Th may not produce the stated bias when low-resolution detectors are used with the computational method listed in Annex A2.

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.6 This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

#### 2. Referenced Documents

2.1 ASTM Standards: <sup>2</sup>

- C 1030 Test Method for Determination of Plutonium Isotopic Composition by Gamma-Ray Spectrometry
- C 1490 Guide for the Selection, Training and Qualification of Nondestructive Assay (NDA) Personnel

C 1592 Guide for Nondestructive Assay Measurements C26.10 Terminology Guide

#### 2.2 ANSI Standard:

N42.14 Calibration and Use of Germanium Spectrometers for the Measurement of Gamma-Ray Emission Rates of Radionuclides<sup>3</sup>

## 3. Terminology

3.1 For definitions of terms used in this test method, refer to Terminology C26.10.

## 4. Summary of Test Method

4.1 The test method consists of measuring the emission rate of 185.7 keV gamma-rays from an item in a controlled geometry and correlating that emission rate with the enrichment of the uranium contained in the item.

4.2 Calibration is achieved using reference materials of known enrichment. Corrections are made for attenuating materials present between the uranium-bearing material and the detector and for chemical compounds different from the calibration reference materials used for calibration.

4.3 The measured items must completely fill the field of view of the detector, and must contain a uranium-bearing material which is infinitely thick with respect to the 185.7 keV gamma-ray. If the field of view is not filled, a correction factor must be applied.

## 5. Significance and Use

5.1 The enrichment meter principle provides a nondestructive measurement of the <sup>235</sup>U fraction of uranium-bearing items. Sampling is not required and no waste is generated, minimizing exposure to hazardous materials and resulting in reduced sampling error.

5.2 This method relies on a fixed and controlled geometry. The uranium-bearing materials in the measured items and calibration reference materials used for calibration must fill the detector field of view.

5.3 Use of a low resolution detector (for example, NaI detector) to measure uranium with  $^{235}$ U fraction approximately 10 % which is contained in a thin-walled container can provide

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<sup>&</sup>lt;sup>2</sup> For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>&</sup>lt;sup>3</sup> Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

a rapid (typically 100 s), easily portable measurement system with precision of 0.6 % and bias of less than 1 %.

5.4 Use of a high resolution detector (for example, highpurity germanium) can provide measurement with a precision better than 0.2 % and a bias less than 1 % within a 300-s measurement time when measuring uranium with  $^{235}$ U fraction in the range of 0.711 % or above which is contained in thin-walled containers.

5.5 In order to obtain optimum results using this method, the chemical composition of the item must be well known, the container wall must permit transmission of the 185.7 keV gamma-ray, and the uranium-bearing material within the item must be infinitely thick with respect to the 185.7 keV gamma-ray. All items must be in identical containers or must have a known container wall thickness and composition.

5.6 Items to be measured must be homogeneous with respect to both  $^{235}$ U fraction and chemical composition.

5.7 When measuring items, using low-resolution detectors, in thin-walled containers that have not reached secular equilibrium (more than about 120 days after processing), either the method should not be used, additional corrections should be made to account for the age of the uranium, or high-resolution measurements should be performed.

5.8 The method is often used as a enrichment verification technique.

## 6. Interferences

6.1 Appropriate corrections must be made for attenuating materials present between the uranium-bearing material and the detector. Inappropriate correction for this effect can result in significant biases.

6.2 Incorrect knowledge of chemical form of the uraniumbearing materials can result in a bias.

6.3 Depending on the dead-time correction method used, excessive dead time can cause errors in live time correction and, thus, result in a measurement bias. Excessive dead time can usually be eliminated by modifications to the detector collimator and aperture.

6.4 Background gamma-rays near 185.7 keV can result in a bias. Table 1 is a list of interfering gamma-rays which may cause an interference.

6.5 Any impurities present in the measured items must be homogeneously distributed and well characterized. The presence of impurities, at concentrations which can measurably attenuate the 185.7 keV gamma-ray and which are not accounted for will result in a bias.

Isotope	Parent	Gamma-Ray Energy (keV)	Measurement Affected
<sup>226</sup> Ra	N/A	185.9	High Resolution, Low Resolution
<sup>212</sup> Pb	<sup>232</sup> U	238.6	Low Resolution
<sup>224</sup> Ra	<sup>232</sup> U	241.0	Low Resolution
<sup>233</sup> Pa	<sup>237</sup> Np	300.1	Low Resolution
<sup>233</sup> Pa	<sup>237</sup> Np	311.9	Low Resolution
<sup>234</sup> Th	<sup>238</sup> U	Bremsstrahlung	Low Resolution
<sup>99</sup> Tc	N/A	Bremsstrahlung	Low Resolution

TABLE 1 Interfering Gamma-Rays

6.6 The presence of radioactive impurities can affect the determination of the 185.7 keV peak area. This type of interference is most often encountered in low-resolution measurement, but can affect high-resolution measurements.

6.7 Other factors, such as the paint on the outside of the cylinders and the condition of the cylinder inner walls after exposure to  $UF_6$ , may affect the precision and bias for both the NaI and the HPGe measurement methods.

#### 7. Apparatus

7.1 *Gamma-Ray Detector System*—General guidelines for selection of detectors and signal-processing electronics are discussed in Guide C 1592, Test Method C 1030, and ANSI standard N42.14. Refer to the References section for a list of other recommended references (1).<sup>4</sup> This system typically consists of a gamma-ray detector, spectroscopy grade amplifier, high-voltage bias supply, multi-channel analyzer, and detector collimator. The system may also include detector backshielding, an ultrasonic thickness gauge, an oscilloscope, a spectrum stabilizer, a computer, and a printer.

7.2 A high-resolution detector system or a low-resolution detector system should be selected, depending on precision and bias requirements for the measurements. Additional detector selection considerations are measurement time, cost, and ease of use. High-resolution detector systems are generally larger, heavier, and more costly than low-resolution detector systems. In addition, the cost of high-resolution detectors is significantly higher (roughly an order of magnitude) than the cost of low-resolution detectors. High-resolution systems, however, provide better results than low-resolution systems, and eliminate some interferences.

7.2.1 *High-Resolution Detector*—A high-resolution detector with a resolution of 1200 eV or better, full width at half maximum, at 122 keV is recommended. Either a planar or coaxial detector can be used, although excessive dead time can result if a coaxial detector with high (>15 %) efficiency is used. The selected detector should be of sufficient size (including a combination of surface area and thickness) to provide the desired counting-statistics based uncertainty within a reasonable counting time.

7.2.2 *Low-Resolution Detector*—A low-resolution detector with the following specifications is recommended: a 5-cm diam, 1.25-cm thick or larger detector with a resolution of 15 % or better at 122 keV.

7.2.3 Collimator and Shield Assembly—The detector collimator and shield assembly must be of sufficient thickness to attenuate in excess of 99.9 % of the 185.7 keV gamma-rays incident upon it. The detector collimator must also block in excess of 99.9 % of the gamma-rays incident upon it and the aperture must restrict the field of view of the detector so that the uranium in the measured items and calibration reference materials used for calibration completely fill the detector field of view. A filter (typically fabricated from cadmium or tin) may, optionally, be included to reduce the intensity of gammainduced X rays from the collimator and shield assembly.

<sup>&</sup>lt;sup>4</sup> The boldface numbers in parentheses refer to the list of references at the end of this standard.

## 7.3 Preparation of Apparatus:

7.3.1 Setup apparatus and set parameters according to manufacturer instructions or site operating procedures.

### 8. Hazards

8.1 Gamma-ray detectors may use power-supply voltages as high as 5 kV. Appropriate precautions should be taken when using, assembling, and disassembling these systems.

8.2 Collimators and shielding may use materials (for example, lead and cadmium) which are considered hazardous and/or toxic and can be physically heavy and difficult to maneuver. Proper care in their use and disposal are required.

8.3 Uranium-bearing materials present both chemical and radiological hazards. The analyst should be aware of these hazards and take appropriate precautions.

## 9. Calibration

9.1 Two types of reference materials are typically used for performing calibration measurements: (1) certified reference materials, and (2) secondary reference materials. Containers in the same configuration as the items to be measured are preferred.

9.1.1 Certified reference materials are commercially available which have been fabricated for the primary purpose of calibration of gamma-ray systems for enrichment measurements using the enrichment meter principle.

9.1.2 Secondary reference materials can be fabricated by analyzing for enrichment using destructive analysis techniques which have been calibrated with a traceable reference material.

9.2 Fill the field of view for the collimated detector, with the uranium in the reference material.

9.3 Measure the reference material for a sufficient amount of time to obtain the desired precision for the net peak area. The precision for the net peak area should be smaller (a factor of ten is recommended) than the target overall measurement system uncertainty.

9.4 Record the identifier for the measured item, the type of uranium-bearing material contained in the item, the counting time used, the net peak area and its uncertainty (or the information needed to compute the net peak area and its uncertainty), and the wall thickness and material. Other information can be recorded as desired. The area for the 185.7 keV peak can be determined using peak fitting or regions of interest. If regions of interest are used to determine the area of the 185.7 keV peak, record the gross counts for each region to be used.

9.5 Repeat steps 9.2-9.4 for other reference materials. The measurement of at least one additional item (total of two) is recommended for calibration of high-resolution systems. The measurement of at least two additional items (total of three) is recommended for calibration of low-resolution systems. If required by regulations, the enrichment of the reference materials used may need to span the range of anticipated enrichments for items to be measured. Use of the method outside the range within which it was calibrated is possible due to the linearity of the calibration, but measurement uncertainty must be considered.

9.6 Determine the calibration constants and their uncertainties using methods shown in Annex A1 and Annex A2, as applicable to the method chosen for peak area determination.

## 10. Procedure

10.1 Good measurement practice includes the measurement of an item used as a control source (refer to Guide C 1592).

10.2 The uranium-bearing material within the measured item must completely fill the field of view of the collimated detector in the geometry used for calibration.

10.3 Precision for the net peak area should be adequate to meet data quality objectives.

10.4 Assess the peak background at the 185.7 KeV measurement environment.

10.5 The area for the 185.7 keV peak must be determined using the same method as was used for calibration (peak fitting or regions of interest). Refer to Table 1 for possible interferences.

10.6 Obtain the wall thickness, and material composition and density for the item's container.

10.7 Document the identifier for the measured item, the chemical form of uranium-bearing material contained in the item, the counting time used, the net peak area and its uncertainty (or the information needed to compute the net peak area and its uncertainty), and the wall thickness and material. Other information can be recorded as desired.

10.8 Compute the attenuation correction factor and its uncertainty using equations shown in Annex A1.

10.9 Use appropriate corrections to account for different chemical forms verses that used during calibration. See References (3) and (4).

10.10 Compute the enrichment and the measurement uncertainty using equations shown in Annex A1 or Annex A2, as appropriate.

#### 11. Precision and Bias

11.1 Precision and bias are dependent on several factors, including (but not limited to): measurement time, accuracy of wall thickness correction factor determination, wall thickness, purity of the measured items, collimation, and calibration uncertainty. In general, the measurement can be tailored to provide the level of precision and bias required. The level of precision is, therefore, typically governed by practical considerations and by the needs of the measurement program and data quality objectives.

11.2 Table 2 demonstrates that the calibration of the method is linear. Using a calibration performed with reference materials ranging in enrichment from 0.31 wt % to 4.46 wt %, containers ranging in enrichment from 12.08 wt % to 97.54 wt % were measured (that is, the instrument was used outside its calibration range). The average bias for this set of measurements was -0.9 % relative. Table 3 demonstrates that this linearity extends to lower enrichments (as low as 0.3206 wt %). A set of measurements performed using a single linear calibration based on measurement of three sources with measured enrichments extending from 0.3206 wt % to 91.419 wt % has an average bias of 0.9 %.

11.3 Table 4 demonstrates that counting statistics is a reasonable predictor of measurement precision for enrichment measurements made using NaI detectors. Ten replicate measurements were made of each of three reference materials.

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## TABLE 2 Measurement of Highly Enriched Uranium in 5A Cylinders (UF<sub>6</sub>) and Z Cans (U<sub>3</sub>O<sub>8</sub>) Using an HPGe Detector (4)

ltem Number	Container Type	Declared <sup>235</sup> U (wt %)	Measured <sup>235</sup> U (wt%) <sup>A</sup>	Difference (wt%)	Rel. Diff. (%)
1	5A	12.08	12.13	0.05	0.4
2	5A	26.44	25.68	-0.76	-2.9
3	5A	44.08	43.28	-0.8	-1.8
4	5A	54.62	55.02	0.4	0.7
5	5A	64.89	64.6	-0.29	-0.4
6	5A	73.21	73.98	0.77	1.1
7	5A	97.60	94.8	-2.77	-2.8
8	Z	22.20	22.51	0.31	1.4
9	Z	33.73	33.41	-0.32	-0.9
10	Z	45.50	44.58	-0.92	-2
11	Z	56.20	57.27	1.07	1.9
12	Z	61.51	58.26	-3.25	-5.3
13	Z	86.57	89.31	2.74	3.2
14	Z	97.54	93.3	-4.24	-4.3
		-0.57	-0.9 <sup>B</sup>		
		1.83	2.5		

<sup>A</sup> Measurement conditions: Items were measured for 100 s each using a planar HPGe detector. Calibration was performed using five certified reference standards ranging in enrichment from 0.31 to 4.46 wt% <sup>235</sup>U and 300 s count times. Nominal wall thickness for 5A cylinders is 0.635 cm of nickel. Nominal wall thickness of Z cans is 0.0381 cm of stainless steel.

<sup>B</sup> Not significant at the 95 % confidence level.

#### TABLE 3 Measurement of Items in Thin-Walled Containers Across a Wide Range of Enrichments Using a Nal Detector

Item Number	Declared Enrichment (% <sup>235</sup> U)	Measured Enrichment (% <sup>235</sup> U) <sup>A</sup>	Difference (wt%)	Rel. Difference (%)
1	0.719	0.681 ± 0.059	-0.038	-5.28
2	0.7258 ± 0.0022	0.700 ± 0.057	-0.0258	-3.55
3	1.962 ± 0.006	2.068 ±0.061	0.106	5.40
4	$3.065 \pm 0.009$	$3.095 \pm 0.064$	0.030	0.98
5	10.200 ± 0.001	10.331 ± 0.085	0.131	1.28
6	11.930 ± 0.036	12.117 ± 0.089	0.187	1.57
7	13.098 ± 0.008	13.040 ± 0.092	-0.058	-0.44
8	17.42 ± 0.052	17.715 ± 0.103	0.295	1.69
9	27	26.930 ± 0.12	-0.07	-0.26
10	37.848 ± 0.015	37.565 ± 0.137	-0.283	-0.75
11	52.426 ± 0.004	52.746 ± 0.164	0.32	0.61
12	66.317 ± 0.032	65.503 ± 0.176	-0.814	-1.23
13	91.419 ± 0.011	89.845 ± 0.204	-1.574	-1.72
14	$0.3206 \pm 0.0002^B$	0.349 ± 0.045	0.0284	8.86
15	0.7209 ± 0.0005 <sup>B</sup>	0.776 ± 0.046	0.0551	7.64
16	1.9664 ± 0.0014 <sup>B</sup>	2.033 ± 0.051	0.0666	3.39
17	2.9857 ± 0.0021 <sup>B</sup>	2.942 ± 0.056	-0.0437	-1.46
18	4.5168 ± 0.0032 <sup>B</sup>	4.450 ± 0.062	-0.0668	-1.48
	-	Average	-0.097	0.847
		Standard Deviation	0.442	3.622

<sup>A</sup> Measurement conditions: Items measured were 1 kg uranium oxide counted for 100 s each replicate. Calibration was performed using three sources (1.962 %, 10.22 %, and 37.848 % <sup>235</sup>U) counted for 300 s each. Nominal wall thickness for items 1 to 13 was 0.02 cm steel. Nominal wall thickness for items 14 to 18 was 0.002 cm aluminum.

<sup>B</sup> Certified reference source.

#### TABLE 4 Replicate Measurement of Three Well-Known Items in Thin-Walled Steel Containers Using an Nal Detector

Reference Value <sup>A</sup>	Number of Replicate Measurements	Average (% <sup>235</sup> U) <sup>B</sup>	Standard Deviation from Replicates (% <sup>235</sup> U)	Standard Deviation Predicted Using Counting Statistics	F <sup>C</sup>
91.419	10	90.806	0.168	0.184	0.83
10.2	10	10.202	0.033	0.072	0.21
1.962	10	1.952	0.063	0.048	1.72

<sup>A</sup> Reference values represent the mean value of destructive analysis results by two independent laboratories.

<sup>B</sup> Measurement conditions: Items measured were 1 kg uranium oxide, in containers with 0.02-cm thick steel walls, counted for 100 s each replicate. Calibration was performed using two sources (3.065 % and 11.93 % <sup>235</sup>U) counted for 300 s each.

 $^{C}$  Significance value for F is 1.88 at (9,  $\infty)$  degrees of freedom and 95 % confidence.

Table 5 demonstrates that counting statistics is also a reasonable predictor of measurement precision for enrichment measurements made using NaI detectors when measuring through steel up to 1.27 cm thick. Ten replicate measurements were made of reference material in two measurement configurations: (1) the reference source alone, and (2) the reference source placed behind 1.27 cm of steel to represent a thick-walled container. Using a single-tailed F test to test for observed



#### TABLE 5 Replicate Measurement of a Well-Known Item With Two Different Wall Thicknesses using an Nal Detector

Reference Value <sup>A</sup>	Number of Replicate Measurements	Average (% <sup>235</sup> U) <sup>B</sup>	Standar Deviation From Replicates (% <sup>235</sup> U)	Standard Deviation Predicted Using Counting Statistics	F <sup>C</sup>
2.95	10	2.98	0.020	0.020	0.98
2.95 <sup>B</sup>	10	2.93	0.035	0.041	0.77

<sup>A</sup> Reference values are New Brunswick Laboratory certified values.

<sup>B</sup> Measurement conditions: Items measured were 200 g uranium oxide in a container with a 0.2 cm aluminum wall, counted for 100 s each replicate. Calibration was performed using two sources (0.71 % and 4.46 % <sup>235</sup>U) counted for 300 s each. The second set of measurements used the same source and detector, but a 1.27 cm thick disc of steel was placed between the source and the detector for both calibration and measurement.

 $^{C}$  Significance value for F is 1.88 at (9, $\infty$ ) degrees of freedom and 95 % confidence.

#### TABLE 6 Measurement of Low Enriched Uranium in 30B (UF<sub>6</sub>) Cylinders using an HPGe Detector (5)

Item	Wall	Declared	Measured	Difference	Rel. Diff.
Inumber	(CIII)	U (WL %)	0 (WI%).	(WL %)	(%)
1	1.23	4.901	4.99	0.08	1.7
2	1.25	2.101	2.10	0.00	0.0
3	1.24	4.900	4.80	-0.10	-2.0
4	1.24	3.908	3.63	-0.28	-7.1
5	1.25	4.403	4.18	-0.22	-5.1
6	1.28	4.405	4.66	-0.25	5.7
7	1.24	3.953	3.75	-0.20	-5.1
8	1.24	3.908	3.93	0.02	0.6
9	1.25	4.405	4.38	-0.03	-0.6
Average		-0.05	-1.3		
Std. Dev.		0.17	4.0		

<sup>A</sup> Measurement conditions: Items were measured for 100 s. each. Calibration was performed using five certified reference standards ranging in enrichment from 0.31 to 4.46 % <sup>235</sup>U and 300 s count times.

	Declared <sup>235</sup> U	Measured <sup>235</sup> U	Difference	Rel. Diff.
Item Number	(wt %)	(wt %) <sup>A</sup>	(wt %)	(%)
1	4.34	4.29	-0.05	-1.2
2	3.95	4.67	0.72	18.2
3	1.60	1.89	0.29	18.1
4	3.91	4.15	0.24	6.1
5	2.88	3.26	0.38	13.2
6	3.95	4.46	0.51	12.9
7	3.62	4.13	0.51	14.1
8	3.40	3.70	0.30	8.5
9	3.38	3.23	-0.15	-4.4
10	3.37	3.09	-0.28	-8.3
11	3.41	3.54	0.13	3.8
12	3.40	3.59	0.19	5.6
13	3.37	3.30	-0.07	-2.1
14	3.40	3.70	0.30	8.8
15	3.82	3.83	0.01	0.3
16	3.82	4.08	0.26	6.8
17	3.82	4.23	0.41	10.7
18	3.81	4.21	0.40	10.5
19	3.40	3.56	0.16	4.7
		Average	0.22	6.7
		Standard Deviation	0.25	7.4

<sup>A</sup> Measurement conditions: Items were measured for 100 s each. Calibration was performed using three 30B Cylinders containing UF<sub>6</sub> for which reference values were obtained from sampling and mass spectrometry. Correction for differences in wall thickness between cylinders was not made. The reference values were: 0.711 %, 3.37, and 4.34 % <sup>235</sup>U. 300 s count times were used for calibration.

standard deviation larger than the standard deviation predicted by counting statistics, neither of the two F values were statistically significant at the 95 % confidence level.

11.4 Field measurement data for UF<sub>6</sub> contained in 30B cylinders are shown in Tables 6 and 7. These data indicate that the performance of the method can be somewhat degraded when measuring through thick-walled containers (30B cylinders have approximately a 1.27 cm thick wall). The standard deviation of the difference for NaI measurements (7.4% relative) is significantly larger than the uncertainty predicted by calibration (2.5% relative). This may result from differences in wall thickness between cylinders. The standard deviation of the

difference for HPGe measurements (4.0 % relative) is also larger than the uncertainty predicted by calibration and propagation of error (1.9 % relative). Because the precision for replicate measurements was demonstrated to be within limits predicted by counting statistics, this indicates that the uncertainty in the correction for the wall thickness may be larger than stated (the estimate used for uncertainty in the wall thickness was based on the uncertainty stated by the ultrasonic thickness gauge manufacturer). Other factors, such as the paint on the outside of the cylinders and the condition of the cylinder inner walls after exposure to UF<sub>6</sub>, may also affect the precision and bias for both the NaI and the HPGe measurement methods.

# TABLE 8 The Effects of Bias Resulting from Drift in Gain from Electronics<sup>A</sup>

Measured Enrichment (wt% 235U)	Ratio (Measured/Reference)		
51.622 ± 0.124	0.985		
51.568	0.984		
51.406	0.981		
51.364	0.980		
51.160	0.976		
50.965	0.972		
50.776	0.969		
50.739	0.968		
50.477	0.963		
50.780	0.969		
Average: 51,086 % <sup>235</sup> U (Reference enrichment: 52,426 wt% <sup>235</sup> U)			

Standard Deviation: 0.394 (about three times the standard deviation predicted by counting statistics)

<sup>A</sup> The 186 keV peak was centered in channel 300 before calibration. During the measurement sequence, the peak drifted 14 channels. Regions of interest were 80 channels wide.

11.5 A series of measurements was taken to demonstrate the potential effect of drift on measurement results for enrichment measurements made using unstabilized NaI detection. Table 8 shows that a significant bias can be introduced by electronic

drift. These measurements were taken indoors (that is, in a somewhat controlled environment); the drift and resultant bias caused by temperature changes experienced in outdoor measurements can be worse than that experienced for these measurements.

11.6 For example, a precision of 0.08 % (relative) has been reported (3) for replicate measurements made on standards. The measurements times needed to attain this level of precision, however, ranged from 10 000 s for a 17 % enriched standard to 277 500 s each (3.21 days) for a standard with 0.3  $\%^{235}$ U. Within relatively short measurement times (300 s), a precision of 0.2 % can be obtained for highly enriched uranium in thin-walled containers.

11.7 As enrichment decreases, however, the precision for the same counting time worsens (e.g., a precision of 3.2 % was obtained for an enrichment of 1.962 % in a thin-walled container using a 300 s measurement time).

#### 12. Keywords

12.1 enrichment; UF<sub>6</sub>; uranium

### ANNEXES

#### (Mandatory Information)

#### A1. CALIBRATION METHOD USING A SINGLE CALIBRATION CONSTANT

A1.1 Calibration can be obtained using a single calibration constant if the net peak area is determined using one of the following two methods:

A1.1.1 Peak and Compton background fitting, or

A1.1.2 Three regions of interest (the 185.7 keV peak and two Compton background regions, one at an energy higher than the 185.7 keV peak, and one at an energy lower than the 185.7 keV peak).

A1.2 The single calibration constant method is typically used for high-resolution gamma-ray systems, but can also be used for low-resolution gamma-ray systems, provided that the peak and Compton background are fit. Resolution is not sufficient for low-resolution detection systems to provide an accurate estimate of net peak area using three regions of interest.

A1.3 When using the single calibration constant method, determine the average calibration constant ( $\overline{K}$ ) and uncertainty

in the calibration factor  $(\sigma_{\overline{K}})$  using the following equations:

$$CF_{i} = e^{\mu_{i}\rho_{i}}$$
(A1.1)  
$$\sigma_{CF_{i}} = CF_{i}\sqrt{\left(t_{i} \times \rho_{i} \times \sigma_{\mu}\right)^{2} + \left(t_{i} \times \mu_{i} \times \sigma_{\rho i}\right)^{2} + \left(\mu_{i} \times \rho_{i} \times \sigma_{i}\right)^{2}}$$

$$P_{i} \times G_{\mu_{i}} + (t_{i} \times \mu_{i} \times G_{\rho i}) + (\mu_{i} \times \mu_{i} \times G_{t_{i}})$$
(A1.2)
$$CR_{i} = CF_{i} \times R_{i}$$
(A1.3)

$$= CF_i \times R_i \tag{A1.3}$$

$$\sigma_{CR_i} = CR_i \sqrt{\left(\frac{\sigma_{CF_i}}{CF_i}\right)^2 + \left(\frac{\sigma_{R_i}}{R_i}\right)^2}$$
(A1.4)

$$\bar{K} = \frac{\sum_{i=1}^{n} \frac{1}{\left(\frac{E_i}{CR_i}\right) \times \left(\left(\frac{\sigma_{E_i}}{E_i}\right)^2 + \left(\frac{\sigma_{CR_i}}{CR_i}\right)^2\right)}}{\sum_{i=1}^{n} \frac{1}{\left(\frac{E_i}{CR_i}\right)^2 \times \left(\left(\frac{\sigma_{E_i}}{E_i}\right)^2 + \left(\frac{\sigma_{CR_i}}{CR_i}\right)^2\right)}}$$
(A1.5)

$$\sigma_{\tilde{K}} = \sqrt{\frac{1}{\sum_{i=1}^{n} \frac{1}{\left(\frac{E_{i}}{CR_{i}}\right)^{2} \times \left(\left(\frac{\sigma_{E_{i}}}{E_{i}}\right)^{2} + \left(\frac{\sigma_{CR_{i}}}{CR_{i}}\right)^{2}\right)}}$$
(A1.6)

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where	
where.	

- $CF_i$  = correction factor for reference material *i*,
- $\mu_i$  = mass attenuation coefficient for the container for reference material *i*,

 $\rho_i$  = density for the container for reference material *i*,

- $t_i$  = thickness of the container for reference material *i*,
- $\sigma_{CFi}$  = uncertainty in the correction factor for reference material *i*,
- $R_i$  = net count rate for reference material *i*,
- $\sigma_{Ri}$  = uncertainty in the net count rate for reference material *i*,
- $CR_i$  = corrected count rate for measurement of reference material *i*,
- $\sigma_{CRi}$  = uncertainty in the corrected count rate for measurement of reference material *i*,
- $E_i$  = enrichment of reference material *i*,
- $\sigma_{Ei}$  = uncertainty in the enrichment for reference material *i*,
- $\bar{K}$  = calibration factor, and
- $\sigma_{\bar{K}}$  = uncertainty in the calibration factor.

A1.4 <sup>235</sup>U fraction and uncertainty for unknown items can

be computed using the following equations:

$$E = \bar{K} \times R \times CF \tag{A1.7}$$

$$\sigma_{E_B} = E \times \left(\frac{\sigma_{\bar{K}}}{\bar{K}}\right) \tag{A1.8}$$

$$\sigma_{E_R} = E \times \sqrt{\left(\frac{\sigma_R}{R}\right)^2 + \left(\frac{\sigma_{CF}}{CF}\right)^2}$$
(A1.9)

where:

- CF = container attenuation correction factor for the unknown,
- $\sigma_{CF}$  = uncertainty in the container attenuation correction factor for the unknown,
- R = net count rate for the unknown,
- $\sigma_R$  = random uncertainty in the net count rate for the unknown,
- $\bar{K}$  = calibration constant,
- $\sigma_{\bar{K}}$  = uncertainty in the calibration constant,
- $E^{n}$  = enrichment of the unknown,
- $\sigma_{E_B}$  = bias estimate from uncertainty in the calibration constant, and
- $\sigma_{E_R}$  = random uncertainty estimate.

### A2. CALIBRATION USING A TWO-CONSTANT CALIBRATION

A2.1 For measurements made using low-resolution detectors, a two-constant calibration is typically used. For this method, two regions of interest are established: (1) the 185.7 keV region, and (2) Compton background at an energy higher than the 185.7 keV region. The following equation is then used:

$$E = A \times R_1 + B \times R_2$$

$$(A2.1)$$

$$\sigma_E = \sqrt{\frac{A^2 R_1 + B^2 \times R_2}{t}}$$

where:

E = measured enrichment,

- $\sigma_E$  = uncertainty in measured enrichment (includes only counting statistics),
- A = scaling constant,

B = scaling constant,

- $R_1$  = count rate (c/s) in the 185.7 keV region,
- $R_2$  = count rate (c/s) in the Compton background region, and
- t = count time (s).

A2.2 The scaling constants, A and B, are estimated from calibration data using a linear regression fit.

$$A = \frac{\sum_{i=1}^{n} \left(\frac{E_{i} \times R_{1_{i}}}{\sigma_{E_{i}}^{2}}\right) \times \sum_{i=1}^{n} \left(\frac{R_{2_{i}}^{2}}{\sigma_{E_{i}}^{2}}\right) - \sum_{i=1}^{n} \left(\frac{E_{i} \times R_{2_{i}}}{\sigma_{E_{i}}^{2}}\right) \times \sum_{i=1}^{n} \left(\frac{R_{1_{i}} \times R_{2_{i}}}{\sigma_{E_{i}}^{2}}\right)}{\sum_{i=1}^{n} \left(\frac{R_{1_{i}}^{2}}{\sigma_{E_{i}}^{2}}\right) \times \sum_{i=1}^{n} \left(\frac{R_{2_{i}}}{\sigma_{E_{i}}^{2}}\right) - \sum_{i=1}^{n} \left(\frac{R_{1_{i}} \times R_{2_{i}}}{\sigma_{E_{i}}^{2}}\right) \times \sum_{i=1}^{n} \left(\frac{R_{1_{i}} \times R_{2_{i}}}{\sigma_{E_{i}}^{2}}\right)}{(A2.2)}$$

$$B = \frac{\sum_{i=1}^{n} \left(\frac{E_{i} \times R_{2_{i}}}{\sigma_{E_{i}}^{2}}\right) \times \sum_{i=1}^{n} \left(\frac{R_{1_{i}}^{2}}{\sigma_{E_{i}}^{2}}\right) - \sum_{i=1}^{n} \left(\frac{E_{i} \times R_{1_{i}}}{\sigma_{E_{i}}^{2}}\right) \times \sum_{i=1}^{n} \left(\frac{R_{1_{i}} \times R_{2_{i}}}{\sigma_{E_{i}}^{2}}\right)}{\sum_{i=1}^{n} \left(\frac{R_{1_{i}}^{2}}{\sigma_{E_{i}}^{2}}\right) - \sum_{i=1}^{n} \left(\frac{R_{1_{i}} \times R_{2_{i}}}{\sigma_{E_{i}}^{2}}\right) \times \sum_{i=1}^{n} \left(\frac{R_{1_{i}} \times R_{2_{i}}}{\sigma_{E_{i}}^{2}}\right)}$$

$$(A2.3)$$

where:

- $E_i$  = enrichment of the measured reference material,
- $\sigma_E$  = uncertainty in the enrichment of the reference material, and
- n = the total number of calibration measurements made.

## A3. ATTENUATION CORRECTION

A3.1 Correction for container wall attenuation is made to obtain accurate results when differences exist between the container wall for the calibration materials and the container wall for measured items, or when there is a significant difference between wall thickness of like items. The difference which can be tolerated before correction is made is dependent on the desired uncertainty in the measurement. Eq A1.1 in Annex A1 is used to compute the attenuation correction factor.

There are three key components of this equation: mass attenuation coefficient, density, and wall thickness. Wall thickness is typically measured for each item using an ultrasonic thickness gauge. Density should be measured using a coupon of the material used to make the container. Although this is rarely done, different alloys and different processes used in making the containers result in metals of sufficient difference in density to make density measurement attractive. Table A3.1 lists mass 🖽 C 1514 – 08

Material	Mass Attenuation Coefficient (cm <sup>2</sup> /g)	Density (g/cm <sup>3</sup> )		
Aluminum	0.122	2.66 – 2.81		
Steel	0.144	7.20 – 7.86		
Stainless Steel	0.144	7.45 -8.02		
Nickel	0.157	8.01 -8.89		
Monel	0.156	8.36 - 8.85		

TABLE A3.1 Mass Attenuation Coefficients and Typical Densities for Selected Materials

attenuation coefficients and typical ranges of density for metals used to make containers commonly used for uranium-bearing materials.

#### **A4. CHEMICAL FORM CORRECTION**

A4.1 For an infinitely thick item the x-ray photo peak rate is inversely proportional to the mass attenuation of the compound.

A4.2 If the calibration is performed with one compound but

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applied to another the correction factor is the ratio of the material attenuation coefficients. In evaluating the material coefficients, coherent scattering is omitted because in and out scatter almost cancel and no energy loss is incurred (9).

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