

## Standard Test Method for Measurement of <sup>235</sup>U Fraction using the 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 ( $\epsilon$ ) 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.

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, from depleted (0.2 %  $^{235}$ U) to very highly enriched (97.5 %  $^{235}$ U).

1.4 Measurement of items that have not reached secular equilibrium between  $^{238}$ U and  $^{234}$ Th, may not produce the stated bias when low-resolution detectors are used with the computational method listed in Appendix B.

1.5 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:

C 982 Standard Guide for Selecting Components for Energy-Dispersive X-Ray Fluorescence (XRF) Systems

#### 3. Terminology

3.1 *Infinite thickness*—The thickness of a material which is at least seven mean free paths for 185.7 keV gamma rays (i.e., the minimum thickness which will attenuate 99.9 % of 185.7 keV gamma rays incident on the compound).

3.2 *Enrichment*—The fraction of  $^{235}$ U relative to total uranium in an item, typically expressed as a weight percentage.

3.3 *Atom Percent*—The ratio of the number of atoms of a given isotope (e.g.,<sup>235</sup>U) to the total number of atoms of an element (e.g., uranium), expressed as a percentage.

3.4 Weight Percent—The ratio of the weight of a given isotope (e.g., <sup>235</sup>U) to the total weight of an element (e.g.,

uranium), expressed as a percentage.

### 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 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.

## 5. Significance and Use

5.1 The enrichment meter principle provides a nondestructive measurement of the  $^{235}$ 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 Use of a low resolution detector (e.g., NaI detector) to measure uranium with  $^{235}$ U fraction approximately 10 % which is contained in a thin-walled container can provide a rapid (typically 100 s), easily portable measurement system with precision of 0.6 % and bias of less than 1 %.

5.3 Use of a high resolution detector (e.g., high-purity germanium) can provide measurement with a precision better than 0.2 % and a bias less than 1 % within a 300-sec measurement time when measuring uranium with  $^{235}$ U fraction in the range of 0.711 % to 4.46 % which is contained in thin-walled containers.

5.4 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.

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

5.6 The uranium-bearing materials in the measured items

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and calibration reference materials used for calibration must fill the detector field of view.

5.7 When measuring items, using low-resolution detectors, in thin-walled containers that have not reached secular equilibrium, 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 typically used as a verification technique, not to establish enrichment.

## 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.

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.

### 7. Apparatus

Gamma-Ray Detector System. General guidelines for selection of detectors and signal-processing electronics are discussed in Guide C 982 and NRC Regulatory Guide 5.9, Rev. 2 (1). This system typically consists of a gamma-ray detector, spectroscopy grade amplifier, high-voltage bias supply, multichannel 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.

7.1 A high-resolution detector system or a low-resolution detector system should be selected, depending on precision and

TABLE 1 Interfering Gamma Rays

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				

bias requirements for the measurements. Additional detector selection considerations are measurement time, cost, and ease of use. Because they are cooled using liquid nitrogen, highresolution detector systems are larger, heavier, and somewhat more difficult to use 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.

7.1.1 High-resolution detector. A high-resolution detector with a resolution of 2000 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.

7.1.2 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.

7.1.3 Other system components. Specifications for amplifier, high-voltage bias supply, multi-channel analyzer, and other components of the system are provided in C 982.

7.1.4 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.

7.2 Preparation of Apparatus

7.2.1 Pole zero. Prior to the use of the detector system, the pole zero should be adjusted per manufacturer's instructions.

7.2.2 Gain. After setting the pole zero, set the gain so that the 185.7 keV peak is well within the observable spectrum. Depending on the software used for analysis, peak position may not be arbitrary.

7.2.3 Other parameters. Other detection parameters (e.g., threshold, zero level) should be configured as described in C 982.

## 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 (e.g., 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) reference materials. Certified reference materials are preferred for calibration of high resolution measurement systems. Containers in the same configuration as the items to be measured are preferred for low resolution measurements.

9.1.1 Certified reference materials are available which have been fabricated for the primary purpose of calibration of gamma-ray systems for enrichment measurements using the enrichment meter principle. These materials are available in the range of <sup>235</sup>U fraction from 0.31 % to 4.46 %. High resolution measurement systems are typically calibrated using these reference materials.

9.1.2 Secondary reference materials can be fabricated by analyzing for enrichment using destructive analysis techniques which have been calibrated with NIST traceable reference materials. Low-resolution measurement systems are typically calibrated using these secondary reference materials.

9.2 Place the container to be used for calibration in contact with the detector collimator, with the uranium in the reference material filling the detector field of view.

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, but is not recommended.

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 Measurement Control. Prior to the measurement of unknown items, local measurement control procedures should be followed. Good measurement practice includes, at a minimum, the measurement of an item used as a control source at the start and end of each day of measurements. The source can be a calibration reference material or an item which is maintained in inventory for the purpose of measurement control, and does not change. The results of these measurements should be evaluated each day of measurement, using a valid statistical technique (e.g., control charting) to assess control.

10.2 Place the detector collimator in contact with the container of the item to be measured, with the item in front of the detector. The uranium-bearing material within the measured item must completely fill the field of view of the detector.

10.3 Measure the item 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 than the target overall measurement system uncertainty.

10.4 Obtain the wall thickness and material type of the item's container.

10.5 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 must be determined using the same method as was used for calibration (peak fitting or regions of interest).

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

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

#### 11. Precision and Bias

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. Because measurement precision is closely related to counting statistics, to a large degree, the measurement technique can be tailored to provide the level of precision and bias required by the measurement program by adjusting the counting time and knowledge of other parameters. 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 sec for a 17 % enriched standard to 277,500 sec each (3.21 days) for a standard with 0.3  $\%^{-235}U.$  Within relatively short measurement times (300 sec), a precision of 0.2 % can be obtained for highly enriched uranium in thinwalled containers. 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 sec measurement time). The level of precision attained is, therefore, typically governed by practical considerations and by the needs of the measurement program.

11.1 Table 2 demonstrates that the calibration of the method is very linear. Using a calibration performed with reference materials ranging in enrichment from 0.31% to 4.46%, containers ranging in enrichment from 12.08% to 97.54% were measured (i.e., the instrument was used outside its

TABLE 2 Measurement of Highly Enriched	Uranium in 5A cylinders (U	$UF_{c}$ ) and Z Cans (U <sub>2</sub> O <sub>2</sub> )	using an HPGe Detector (4)

	Declared <sup>35</sup> U (wt %) <u>12.08</u> <u>26.44</u> <u>44.08</u> <u>54.62</u> <u>64.89</u> <u>73.21</u>	Measured <sup>235</sup> U (wt%) <sup>A</sup> 12.13 25.68 43.28 55.02 64.6 73.98	Difference (wt%) -0.76 -0.8 0.4 -0.29	Rel. Diff. (%) -2.9 -1.8 0.7 -0.4			
1     5A       2     5A       3     5A       4     5A       5     5A       6     5A       7     5A       8     Z       9     Z       10     Z	12.08 26.44 44.08 54.62 64.89 73.21	12.13 25.68 43.28 55.02 64.6	0.05 -0.76 -0.8 0.4 -0.29	0.4 -2.9 -1.8 0.7			
2         5A           3         5A           4         5A           5         5A           6         5A           7         5A           8         Z           9         Z           10         Z	26.44 44.08 54.62 64.89 73.21	25.68 43.28 55.02 64.6	-0.76 -0.8 0.4 -0.29	-2.9 -1.8 0.7			
3     5A       4     5A       5     5A       6     5A       7     5A       8     Z       9     Z       10     Z	44.08 54.62 64.89 73.21	43.28 55.02 64.6	-0.8 0.4 -0.29	-1.8 0.7			
4         5A           5         5A           6         5A           7         5A           8         Z           9         Z           10         Z	54.62 64.89 73.21	55.02 64.6	0.4	0.7			
5         5A           6         5A           7         5A           8         Z           9         Z           10         Z	64.89 73.21	64.6	-0.29				
6         5A           7         5A           8         Z           9         Z           10         Z	73.21			-0.4			
7         5A           8         Z           9         Z           10         Z		73.98					
8         Z           9         Z           10         Z			0.77	1.1			
9 Z 10 Z	97.60	94.8	-2.77	-2.8			
10 Z	22.20	22.51	0.31	1.4			
10 2	33.73	33.41	-0.32	-0.9			
11 7	45.50	44.58	-0.92	-2			
	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			
		Average	-0.57	-0.9 <sup>B</sup>			
	Standard Deviation 1.83 2.5						

<sup>4</sup>Measurement conditions: Items were measured for 100 sec each using a planar HPGe detector. Calibration was performed using five certified reference standards ranging in enrichment from 0.31 to 4.46 % <sup>235</sup>U and 300 sec 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.

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 %). A set of measurements performed using a single linear calibration based on measurement of three sources with measured enrichments extending from 0.3206 % to 91.419 % has an average bias of 0.9 %.

11.2 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.

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 configu-

rations: (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 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.

11.3 Field measurement data for  $UF_6$  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

	TABLE 5 Measurement of items in thin-walled Containers Across a wide Kange of Emittiments Using a Nai Delector.						
Item	Declared	Measured Enrichment	Difference	Rel.			
Number	Enrichment	(% <sup>235</sup> U) <sup>A</sup>	(wt%)	Difference			
	(% <sup>235</sup> U)	· · · · ·		(%)			
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 ± 0.009	3.095 ± 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 ± 0.0002 <sup>B</sup>	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>	$2.942 \pm 0.056$	-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			

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

<sup>A</sup>Measurement conditions: Items measured were 1 kg uranium oxide counted for 100 sec each replicate. Calibration was performed using three sources (1.962 %, 10.22 %, and 37.848 % <sup>235</sup>U) counted for 300 sec 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.

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#### 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 sec each replicate. Calibration was performed using two sources (3.065 % and 11.93 % <sup>235</sup>U) counted for 300 sec each.

<sup>C</sup>Significance value for F is 1.88 at (9,  $\infty$ ) degrees of freedom and 95 % confidence.

#### 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 sec each replicate. Calibration was performed using two sources (0.71 % and 4.46 % <sup>235</sup>U) counted for 300 sec 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.

<sup>*c*</sup>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)

ltem Number	Wall (cm)	Declared <sup>235</sup> U (wt %)	Measured <sup>235</sup> U (wt%) <sup>A</sup>	Difference (wt %)	Rel. Diff. (%)
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>4</sup>Measurement conditions: Items were measured for 100 sec. each. Calibration was performed using five certified reference standards ranging in enrichment from 0.31 to 4.46 % <sup>235</sup>U and 300 sec count times.

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.

11.4 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 (i.e., 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.

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#### TABLE 7 Measurement of Low Enriched Uranium in 30B (UF<sub>6</sub>) Cylinders using a Nal Detector (6)

Item Number	Declared <sup>235</sup> U	Measured <sup>235</sup> U	Difference	Rel. Diff.
	(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 sec 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 sec count times were used for calibration.

TABLE 8	The Effects of	Bias	Resulting	from	Drift	in	Gain	from
		Elec	ctronics. <sup>A</sup>					

Measured Enrichment (% <sup>235</sup> U)	Ratio (Measured/Reference)
$51.622 \pm 0.124$	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
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Average: 51.086 % <sup>235</sup>U (Reference enrichment: 52.426 % <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.

## ANNEXES

#### (Mandatory Information)

#### A1. Calibration Method Using a Single Calibration Constant

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

(1) Peak and Compton background fitting,

(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).

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 reigons of interest.

When using the single calibration constant method, determine the average calibration constant ( $\bar{K}$ ) and uncertainty in the calibration factor ( $\sigma_{\bar{K}}$  using the following equations:

$$CF_i = e^{\mu_i \rho_i t i} \tag{A1.1}$$

$$\sigma_{CF_i} = CF_i \sqrt{(t_i \times \rho_i \times \sigma_{\mu_i})^2 + (t_i \times \mu_i \times \sigma_{\rho_i})^2 + (\mu_i \times \rho_i \times \sigma_{t_i})^2}$$
(A1.2)

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

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

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

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

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

where:

- $CF_i$ = Correction factor for reference material i
- = Mass attenuation coefficient for the container for  $\mu_i$ reference material i
- = Density for the container for reference material i  $\rho_i$
- = Thickness of the container for reference material i t;
- = Uncertainty in the correction factor for reference  $\sigma_{CFi}$ material i
- $R_i$ = Net count rate for reference material i
- = Uncertainty in the net count rate for reference  $\sigma_{Ri}$ material i
- $CR_i$ = Corrected Count rate for measurement of reference material i
- = Uncertainty in the corrected count rate for mea- $\sigma_{CRi}$ surement of reference material i

- $E_i$ = Enrichment of reference material i
- = Uncertainty in the enrichment for reference mate- $\sigma_{Ei}$ rial i
- = Calibration factor and Ā
- = Uncertainty in the calibration factor  $\sigma_{\bar{K}}$

<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
- = Random uncertainty in the net count rate for the  $\sigma_R$ unknown
- Ā = Calibration constant
- = Uncertainty in the calibration constant  $\sigma_K$
- Ε = Enrichment of the unknown
- $\sigma_{E_B}$ = Bias estimate from uncertainty in the calibration constant and
- = Random uncertainty estimate  $\sigma_{E_P}$

#### A2. Calibration Using a Two-Constant Calibration

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

Where

- Ε = Measured enrichment
- $\sigma_E$  = Uncertainty in measured enrichment (includes only counting statistics)
- = Scaling constant A
- = Scaling constant В
- $R_1$  = 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)

The scaling constants, A and B, are estimated from calibra-

tion data using a linear regression fit.

#### Where

- $E_i$  = Enrichment of the measured reference material
- $\sigma_F$  = Uncertainty in the enrichment of the reference material and

= the total number of calibration measurements made. n

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

$$B = \frac{\sum_{i=1}^{n} \left(\frac{E_{i} \times R_{2_{i}}}{\sigma_{E_{i}}^{2}}\right) \times \sum_{i=1}^{n} \left(\frac{R_{1_{i}}^{2}}{\sigma_{E_{i}}^{2}}\right) - \sum_{i=1}^{n} \left(\frac{E_{i} \times R_{1_{i}}}{\sigma_{E_{i}}^{2}}\right) \times \sum_{i=1}^{n} \left(\frac{R_{1_{i}} \times R_{2_{i}}}{\sigma_{E_{i}}^{2}}\right)}{\sum_{i=1}^{n} \left(\frac{R_{1_{i}}^{2}}{\sigma_{E_{i}}^{2}}\right) \times \sum_{i=1}^{n} \left(\frac{R_{2_{i}}^{2}}{\sigma_{E_{i}}^{2}}\right) - \sum_{i=1}^{n} \left(\frac{R_{1_{i}} \times R_{2_{i}}}{\sigma_{E_{i}}^{2}}\right)^{2}}$$
(A2.3)

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#### A3. Attenuation Correction

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 A3.1lists mass attenuation coefficients and typical ranges of density for metals used to make containers commonly used for uranium-bearing materials.

TABLE A3.1	Mass Attenuation	Coefficients	and 1	Typical Densit	lies
	for Select	ted Materials	5		

Material	Mass Attenuation Coefficient (cm <sup>2</sup> /g)	Density (g/cm <sup>3</sup> )
		(0 /
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

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