



Designation: C1514 – 08

Standard Test Method for Measurement of ^{235}U Fraction Using Enrichment Meter Principle¹

This standard is issued under the fixed designation C1514; 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 (ϵ) 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 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 ^{238}U and ^{234}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:*²

- C1030 Test Method for Determination of Plutonium Isotopic Composition by Gamma-Ray Spectrometry
- C1490 Guide for the Selection, Training and Qualification of Nondestructive Assay (NDA) Personnel

¹ This test method is under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.10 on Non Destructive Assay.

Current edition approved July 1, 2008. Published August 2008. Originally approved in 2002. Last previous edition approved in 2002 as C1514 – 02. DOI: 10.1520/C1514-08.

² 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.

C1592 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 Radionuclides³

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 ^{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 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.

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

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 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, high-purity 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 an 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 uranium-bearing 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.

TABLE 1 Interfering Gamma-Rays

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

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.

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 **C1592**, Test Method **C1030**, and ANSI standard N42.14. Refer to the References section for a list of other recommended references (1).⁴ 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

⁴ The boldface numbers in parentheses refer to the list of references at the end of this standard.

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 gamma-induced X rays from the collimator and shield assembly.

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 [C1592](#)).

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 versus 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

TABLE 2 Measurement of Highly Enriched Uranium in 5A Cylinders (UF₆) and Z Cans (U₃O₈) Using an HPGe Detector (4)

Item Number	Container Type	Declared ²³⁵ U (wt %)	Measured ²³⁵ U (wt%) ^A	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	
				Average	-0.57	-0.9 ^B
				Standard Deviation	1.83	2.5

^A 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% ²³⁵U 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.

^B Not significant at the 95 % confidence level.

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. **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 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 UF₆ 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

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

Item Number	Declared Enrichment (% ²³⁵ U)	Measured Enrichment (% ²³⁵ U) ^A	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 ± 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 ^B	0.349 ± 0.045	0.0284	8.86	
15	0.7209 ± 0.0005 ^B	0.776 ± 0.046	0.0551	7.64	
16	1.9664 ± 0.0014 ^B	2.033 ± 0.051	0.0666	3.39	
17	2.9857 ± 0.0021 ^B	2.942 ± 0.056	-0.0437	-1.46	
18	4.5168 ± 0.0032 ^B	4.450 ± 0.062	-0.0668	-1.48	
			Average	-0.097	0.847
			Standard Deviation	0.442	3.622

^A 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 % ²³⁵U) 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.

^B Certified reference source.