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# Standard Test Method for
# Nondestructive Assay of Special Nuclear Material in Low-Density Scrap and Waste by Segmented Passive Gamma-Ray Scanning[1]

This standard is issued under the fixed designation C1133; 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 transmission-corrected nondestructive assay (NDA) of gamma-ray emitting special nuclear materials (SNMs), most commonly $^{235}$U, $^{239}$Pu, and $^{241}$Am, in low-density scrap or waste, packaged in cylindrical containers. The method can also be applied to NDA of other gamma-emitting nuclides including fission products. High-resolution gamma-ray spectroscopy is used to detect and measure the nuclides of interest and to measure and correct for gamma-ray attenuation in a series of horizontal segments (collimated gamma detector views) of the container. Corrections are also made for counting losses occasioned by signal processing limitations (**1-3**).[2]

1.2 There are currently several systems in use or under development for determining the attenuation corrections for NDA of radioisotopic materials (**4-8**). A related technique, tomographic gamma-ray scanning (TGS), is not included in this test method (**9,10**).

1.2.1 This test method will cover two implementations of the Segmented Gamma Scanning (SGS) procedure: (*1*) Isotope Specific (Mass) Calibration, the original SGS procedure, uses standards of known radionuclide masses to determine detector response in a mass versus corrected count rate calibration that applies only to those specific radionuclides for which it is calibrated, and (*2*) Efficiency Curve Calibration, an alternative method, typically uses non-SNM radionuclide sources to determine system detection efficiency vs. gamma energy and thereby calibrate for all gamma-emitting radionuclides of interest (**11**). These two methods will be covered in detail in the remainder of the main body of this test method and Annex A1.

1.2.1.1 Efficiency Curve Calibration, over the energy range for which the efficiency is defined, has the advantage of providing calibration for many gamma-emitting nuclide for which half-life and gamma emission intensity data are available.

1.3 The assay technique may be applicable to loadings up to several hundred grams of nuclide in a 208-L (55-gal) drum, with more restricted ranges to be applicable depending on specific packaging and counting equipment considerations.

1.4 Measured transmission values must be available for use in calculation of segment-specific attenuation corrections at the energies of analysis.

1.5 A related method, SGS with calculated correction factors based on sample content and density, is not included in this standard.

1.6 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.7 *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.* Specific precautionary statements are given in Section 8.

## 2. Referenced Documents

2.1 *ASTM Standards:*[3]

C982 Guide for Selecting Components for Energy-Dispersive X-Ray Fluorescence (XRF) Systems[4]

C1030 Test Method for Determination of Plutonium Isotopic Composition by Gamma-Ray Spectrometry

C1128 Guide for Preparation of Working Reference Materials for Use in Analysis of Nuclear Fuel Cycle Materials

C1156 Guide for Establishing Calibration for a Measurement Method Used to Analyze Nuclear Fuel Cycle Materials

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[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 Nondestructive Assay.

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[2] The boldface numbers in parentheses refer to the list of references at the end of this test method.

[3] 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.

[4] Withdrawn. The last approved version of this historical standard is referenced on www.astm.org.

C1207 Test Method for Nondestructive Assay of Plutonium in Scrap and Waste by Passive Neutron Coincidence Counting

C1210 Guide for Establishing a Measurement System Quality Control Program for Analytical Chemistry Laboratories Within the Nuclear Industry

C1458 Test Method for Nondestructive Assay of Plutonium, Tritium and [241]Am by Calorimetric Assay

E181 Test Methods for Detector Calibration and Analysis of Radionuclides

2.2 *ANSI Standards:*[5]

ANSI/IEEE 325 Test Procedures for Germanium Gamma-Ray Detectors

ANSI N42.14 Calibration and Use of Germanium Spectrometers for the Measurement of Gamma-Ray Emission Rates of Radionuclides

2.3 *NRC Regulatory Guides:*[6]

Regulatory Guide 5.9, Rev. 2, Guidelines for Germanium Spectroscopy Systems for Measurement of Special Nuclear Materials

Regulatory Guide 5.11, Rev. 1, Nondestructive Assay of Special Nuclear Material Contained in Scrap and Waste

Regulatory Guide 5.53, Rev. 1, Qualification, Calibration, and Error Estimation Methods for Nondestructive Assay

## 3. Summary of Test Method

3.1 The assay of the nuclides of interest is accomplished by measuring the intensity of one or more characteristic gamma rays from each nuclide. Corrections are made for count rate-related losses and attenuation by the item. The appropriate mass or efficiency calibration then provides the relationship between observed gamma-ray intensity and nuclide content.

3.2 Either of two distinct calibration methods can be used:

3.2.1 *Isotope Specific Calibration* provides assay results for only those radionuclides for which the SGS is specifically calibrated. Calibration is performed using standards containing the radionuclides to be assayed.

3.2.2 *Efficiency Curve Calibration* entails determination of the system detecton efficiency as a function of gamma ray energy. Analysis of assay data consists of using the energy of a peak to infer the emitting radionuclide, and then calculating the radionuclide mass from the specific activity and the gamma emission intensity of the radionuclide, and the corrected count rate and detector efficiency at the peak energy.

3.3 The assay item is rotated about its vertical axis and scanned segment by segment along that axis, thereby reducing the effects of nonuniformity in both matrix density and nuclide distribution (see Fig. 1).

3.4 Count rate-dependent losses from pulse pile-up and analyzer dead time are corrected for by electronic modules, a radioactive source, a pulser, or a combination of these.

3.5 The average linear attenuation coefficient of each horizontal segment is calculated by measurement of the transmitted intensity of an appropriate external gamma-ray source. The
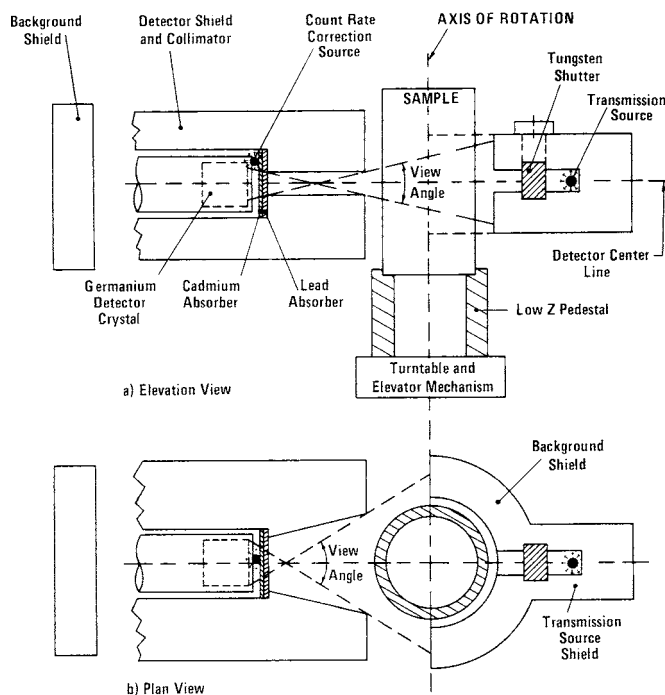


FIG. 1 Typical Arrangement for Segmented Gamma-Ray Scanning

source is mounted directly opposite the gamma-ray detector, on the far side of the assay item (see Fig. 1).

3.6 Two conditions must be met to optimize SGS assay results as follows:

3.6.1 The particles containing the nuclides of interest must be small enough to minimize self-absorption of emitted gamma radiation (**12**).

3.6.1.1 Under specific conditions, particles large enough to provide significant self absorption (lumps ) may be assayed accurately. These conditions include use of specific Nuclide differential peak calibration and calibration using mass standards that have the same attenuation characteristics over the energy range used for quantitative measurements as the materials to be assayed.

3.6.1.2 An alternative approach to mass calibration with standards that contain the same sized particles is to apply correction algorithms that are based on the differential response of two or more peaks at different energies from the same nuclide. For example, the 129 and 414 keV peaks of [239]Pu or the 144 and 186 keV peaks of [235]U could be used (see 6.7).

3.6.1.3 The presence of lumps in material being assayed also can be detected using differential peak response algorihms.

3.6.2 The mixture of material within each item segment must be sufficiently uniform to apply an attenuation correction factor, generally computed from a measurement of gamma-ray transmission through the segment.

3.7 The corrected gamma-ray count rates for the nuclides of interest are determined on a segment-by-segment basis. The precision of the measured count rate of each gamma ray used for analysis is also estimated on a segment-by-segment basis. At the completion of the measurement of all segments, corrected count rates are summed, and mass values for the nuclides of interest in the entire container are calculated based

either on comparisons to appropriate calibration materials or from the gamma emission rates determined from the segment efficiencies determined over the energy range of interest. Based on counting statistics for individual segments, precision values are propagated to obtain the estimated precision of the analysis.

3.8 In the event that a single nuclide of an element is measured and the total element mass is required (for example, $^{239}$Pu and total plutonium), it is common practice to apply a known or estimated nuclide/total element ratio to the nuclide assay value to determine the total element content.

3.8.1 Isotope ratios can be determined using gamma isotopic analysis techniques such as those described in Test Method C1030.

## 4. Significance and Use

4.1 Segmented gamma-ray scanning provides a nondestructive means of measuring the nuclide content of scrap and waste where the specific nature of the matrix and the chemical form and relationship between the nuclide and matrix may be unknown.

4.2 The procedure can serve as a diagnostic tool that provides a vertical profile of transmission and nuclide concentration within the item.

4.3 Sample preparation is generally limited to good waste/ scrap segregation practices that produce relatively homogeneous items that are required for any successful waste/ inventory management and assay scheme, regardless of the measurement method used. Also, process knowledge should be used, when available, as part of a waste management program to complement information on sample parameters, container properties, and the appropriateness of calibration factors.

## 5. Interferences

5.1 Radionuclides may be present in the assay item that produce gamma rays with energies that are the same or very nearly the same as the gamma rays suggested for nuclide or transmission measurement. The areas of the closely spaced peaks that are produced in the gamma-ray spectrum cannot be calculated by simple spectroscopic procedures. Peak fitting software routines may be able to resolve closely spaced peaks in some cases; if not, the nuclide of interest may produce other gamma rays that may be used for analysis.

5.1.1 The peak produced by the 661.6-keV gamma ray from $^{137}$Cs would interfere with calculation of the area of the $^{241}$Am peak produced by its 662.4-keV gamma ray. The 721.9-keV gamma ray of $^{241}$Am may be a useful alternative.

5.1.2 The peak produced by the 765.8-keV gamma ray from $^{95}$Nb would interfere with calculation of the area of the $^{238}$Pu peak produced by its 766.4-keV gamma ray. The 786.3-keV gamma ray of $^{238}$Pu may be a useful alternative.

5.1.3 Occasionally, $^{237}$Np is found in the presence of plutonium. The $^{237}$Np daughter, $^{233}$Pa, emits a gamma ray at 415.8-keV along with several gamma rays in the range from 300 to 400 keV. Peaks from these gamma rays would interfere with calculation of the area of the $^{239}$Pu peak produced by its 413.7-keV gamma ray and several other often used peaks from $^{239}$Pu. In this case, the peak produced by the 129.3-keV gamma ray of $^{239}$Pu may be the only reasonable alternative.

5.1.4 The peak produced by the 63.1-keV gamma ray from $^{169}$Yb, sometimes used as the transmission source for $^{235}$U assays, may interfere with calculation of the area of the peak produced by the 59.5-keV gamma ray of $^{241}$Am, which is used as the count rate correction source. The $^{169}$Yb gamma ray can be sufficiently attenuated by placing a cadmium absorber over the transmission source or the problem can be avoided altogether by using a two-pass assay. The first measurement pass measures the intensity of the transmission source for each segment. The second measurement pass measures the intensity of the 413.7-keV $^{239}$Pu gamma-ray emission from each segment with the transmission source shutter closed.

5.1.5 Transmission source peaks may have errors introduced by the presence of a radionuclide in the assay material that emits gamma rays at or near one or more of the measured transmission energies. The affected measurements will then be higher than the actual transmissions through the item, leading to calculation of a lower than actual correction factor and quantity of measured radionuclide.

5.2 In the case of $^{239}$Pu assays using $^{75}$Se as a transmission source, random coincident summing of the 136.00 and 279.53-keV gamma-ray emissions from $^{75}$Se produces a low-intensity peak at 415.5-keV that interferes with calculation of the area of the $^{239}$Pu peak produced by its 413.7-keV gamma ray. The effects of this sum-peak can be reduced by attenuating the radiation from the transmission source to the lowest intensity required for transmission measurements of acceptable precision. This problem also can be avoided by making a two-pass assay.

5.3 Peaks may appear at the gamma-ray energies used for analysis when there is no nuclide present on the turntable. The likely cause is excessive amounts of nuclide stored in the vicinity of the detector. The preferred solution to this problem is removal of the nuclide from the vicinity and restraint of nuclide movements around the system during measurements. If these conditions cannot be met, sufficient shielding must be provided to eliminate these peaks. Shielding opposite the detector, on the far side of the item to be assayed, will also help to reduce the amount of ambient radiation seen by the detector (see Fig. 1).

## 6. Sources of Error

6.1 Sources of error specifically applicable to segmented gamma-ray scanning are discussed in this section. General descriptions of sources of error encountered in gamma-ray nondestructive assay systems can be found in NRC Regulatory Guide 5.11.

6.2 The bias in an assay is strongly dependent on how well the attenuation for each segment has been determined. In order to determine the attenuation, a radioactive source with a gamma ray of nearly the same energy as the gamma ray of the nuclide of interest is positioned directly opposite the gamma-ray detector, on the far side of the assay item (see Table 1 for suggested nuclide/transmission source combinations and Fig. 1 for geometry). At lower energies, where the mass attenuation coefficient varies rapidly, it is useful to find a source that produces gamma rays with energies that bracket the energy of the gamma ray from the nuclide of interest. This test method

**TABLE 1 Suggested Nuclide/Source Combinations for Segmented Gamma-Ray Assay**

| Nuclide | Peak Energy, keV | Trans-mission Source | Peak Energy, keV | Count Rate Correction Source | Peak Energy, keV |
|---|---|---|---|---|---|
| $^{235}$U | 185.7 | $^{169}$Yb | 177.2, 198.0 | $^{241}$Am | 59.5 |
| $^{238}$U | 1001.1 | $^{54}$Mn | 834.8 | $^{137}$Cs | 661.6 |
| | | $^{60}$Co | 1173.2, 1332.5 | | |
| $^{237}$Np | 311.9 | $^{203}$Hg | 279.2 | $^{235}$U | 185.7 |
| $^{238}$Pu | 766.4 | $^{137}$Cs | 661.6 | $^{133}$Ba | 356.3 |
| $^{239}$Pu | 413.7, 129.3 | $^{75}$Se | 400.1 | $^{133}$Ba | 356.3 |
| $^{241}$Am | 662.4 | $^{75}$Se | 400.1 | $^{133}$Ba | 356.3 |

provides a more accurate procedure for calculation of attenuation at the energy of interest. At higher energies, where the mass attenuation coefficient varies more slowly, a transmission source with a single gamma ray of nearly the same energy as the nuclide of interest may provide a sufficiently accurate determination of attenuation.

6.3 Radionuclides emitting low-energy radiation, especially $^{241}$Am, may contribute a large fraction of the total count rate. The low-energy radiation may be reduced by the use of fixed absorbers, typically cadmium, tin, or lead, between the asay item and the detector (see Fig. 1 and 7.2.7).

6.4 Radionuclides emitting high-energy radiation will contribute Compton-continuum under peaks to be used for the assay. The Compton-continuum will worsen the estimated precision calculated from the counting statistics. The assay of $^{235}$U is normally performed using $^{169}$Yb as the transmission source. This source provides 177- and 198-keV gamma rays that allow accurate calculation of the transmission at 185.7-keV, the energy of the gamma ray from $^{235}$U normally used for assays. The problem of added Compton-continuum from the Yb source can be avoided by making a two-pass assay. If the high-energy gamma rays are from the assay item itself, but not from the nuclide of interest, it may be possible to eliminate them from future assay items by scrap and waste segregation procedures. Such procedures are discussed in detail in NRC Regulatory Guide 5.11.

6.5 If the transmission source nuclide or a radionuclide with one or more gamma rays of similar energy is in the assay material, a two-pass assay allows the passive scan data to be used as the background for the transmission measurement.

6.6 Variations in item composition and density within a segment lead to indeterminate errors. Such variations should be minimized through appropriate scrap and waste segregation procedures.

6.7 Some matrix forms may be unsuitable for segmented gamma-ray analysis procedures.

6.7.1 Such forms may contain lumps of nuclide, that is, nuclide contained in small volumes having a localized density substantially different from the bulk density of the rest of the container. The dimensions of nuclide particles that constitute a lump vary with the energy of the emitted radiation used for the analytical measurement. The possible magnitude of the problem may be estimated from examples of attenuating effects provided in Note 1.

NOTE 1—A plutonium metal sphere 0.02 cm in diameter will absorb approximately 4 % of the 414-keV $^{239}$Pu gamma rays produced. Approxi-

mately 15 % of the 186-keV $^{235}$U gamma rays will be absorbed in a uranium metal sphere of the same diameter (12).

6.7.2 The presence of lumps of plutonium may be detected and, in some cases, a corrected value calculated using various correction algorithms. The techniques use transmission-corrected assay results for multiple gamma-ray energies from a single nuclide and a weighting function to account for self-absorption by lumps. This approach has been used primarily for the analysis of $^{239}$Pu, where the nuclide of interest s emits gamma rays over a range of several hundred keV. The success of the lump correction calculations is not universal (6-8), however, and the technique must be evaluated for specific material streams prior to implementation.

6.7.3 Another condition that will cause measurement problems is presented by containers with radically heterogeneous contents having highly variable densities and non-uniform activity distributions, that prevent the calculation of a valid attenuation correction based on the transmission measurement. In the case of such a condition, an analytical method less sensitive to nuclide and matrix densities should be used.

6.8 The nature of the segmenting process leads to end effect problems. During counting, the detector's field of view in the vertical direction is larger than the horizontal extensions of the top and bottom planes of the collimator (see Fig. 1). Throughout most of the item, the results of this overview present no particular problem since calibration procedures effectively account for it. However, the top and bottom segments present particular problems. If the limits of the scan are set to match the top and bottom of the item to straight line extensions of the collimator's top and bottom planes, the nuclide material in the top and bottom segments is viewed for a period of time 65 to 80 as long as nuclide toward the center of the assay item. Scanning beyond the end of the item is likely to overestimate the nuclide content of the bottom segment due to the high density of the turntable itself and underestimate the nuclide content of the top segment as the detector looks over the top of the item. One way to decrease this problem involves the placement of a hollow cylindrical pedestal with high transmission between the item and the turntable (see Fig. 1), combined with scanning beyond the end of the item on both ends. Another option, more difficult to implement, involves the previous two steps along with application of the measured attenuation from the nearest item segment, to the appropriate, overscanned segments (1,7).

## 7. Apparatus

7.1 The following considerations apply specifically to segmented gamma-ray scanners. General guidelines for the selection of detectors and signal processing electronics are discussed in Guide C982 and NRC Regulatory Guide 5.9. Data acquisition systems are considered in NRC Regulatory Guide 5.9.

7.2 Complete hardware and software systems for high-resolution, segmented gamma-ray scanning of both large and small items of waste and scrap containing SNM are commercially available. It is recommended that the system have the following components:

7.2.1 *Germanium Detector*, with appropriate electronics to handle the required count rates. A wide range of Germanium

crystal volumes are available. Crystal dimensions determine the efficiency of the detector. Detectors with efficiencies ranging over more than an order of magnitude are available as standard products. Detectors with resolutions better than 850 eV full width at half maximum at 122-keV ($^{57}$Co) are recommended. Test procedures for detectors are given in Test Methods E181, ANSI/IEEE 325, and ANSI N42.14.

7.2.2 *Computer*—Computer appropriate for control of the assay hardware, performance of analysis computations, and display and storage of the data and results.

7.2.3 *Motorized, Vertical Scanning Turntable*—Turntable capable of accommodating the largest size and weight containers to be measured is required. For normal analyses, segment sizes between the height of the collimator and one-half the collimator height provide sufficient segmentation. The system should provide acceptable detector-assay item positioning accuracy and repeatability (± 0.5 % of the range of travel is commercially available). Both helical or fixed-segment counting schemes are acceptable, and either the assay item or the detector-collimator and transmission source shield assemblies can be moved. The turntable rotational speed should provide either a large number of rotations (ten or more) or a small integral number of rotations during the counting period for each segment.

7.2.4 *Detector Collimator*—Collimator constructed of lead or tungsten serves to define the detector's horizontal and vertical viewing angles and to shield the detector from ambient radiation. A deep collimator (front to back), along with close coupling of the collimator and assay item, reduces the vertical viewing angle and improves segmentation. The reduced viewing angle decreases the bias of the attenuation correction and decreases the severity of end effects. Collimator slit height should be chosen to be in the range ⅛ to 1/16 of the height of the assay item. The horizontal field of view must include the entire diameter of the item. Lining the inside of the collimator with appropriately-thick sheets of cadmium or tin and copper will eliminate collimator lead X-rays from the spectrum.

7.2.4.1 For large items, where high efficiency is required for reasonable count times, the height of the collimator slit should be approximately equal to the diameter of the detector crystal. In practice, collimator depth/height ratios of two to four for 208-L (55-gal) drum-sized items are reasonable.

7.2.4.2 Smaller items require narrower (vertical) collimators to maintain the benefits of accurate attenuation corrections and to minimize end effects. A collimator depth/height ratio of six to ten is reasonable.

7.2.5 *Count-Rate Correction Source*—Correction source is chosen to have gamma-ray emission energies that are lower than the energy of the gamma ray from the nuclide of interest in order to avoid Compton interferences. These sources can be obtained as 5 to 10 µCi, flat plastic wafer, sealed sources, for easy attachment close to the detector. Recommended sources are listed in Table 1. A combination of cadmium or tin and copper (closest to the detector) foils positioned under the source reduce the effect of abundant low-energy gamma rays that are present with some of the suggested count-rate correction sources. The position of the source is adjusted to produce a count rate providing sufficient precision for the assay times

used and then fixed in place. Alternatively, an electronic pulser can be used for count rate correction.

7.2.6 *Transmission Source*—Transmission source must be considerably stronger than the count-rate correction source to perform effectively. Ten to 50 mCi sources for small item counters and 50 to 100 µCi sources for barrel size counters, in the shape of small diameter rods, are well suited to use in cylindrical lead or tungsten shields. These shields reduce radiation exposure to workers and collimate the radiation from the transmission source to a narrow region containing the detector. If an assay system is to be calibrated for multiple radionuclides, it may be useful to select a transmission source having multiple gamma ray energies (with appropriate relative intensities), and use a suitable method to determine transmissions at the radionuclide analysis energies. Table 1 provides a listing of suggested nuclides for use as transmission sources, with the listed nuclides of interest. Because an otherwise appropriate source isotope can be relatively short-lived, it may be necessary to obtain one with an activity considerably above the optimum to provide for a useful working life. The count rate of a new source may be attenuated by collimation, absorbers directly in front of the source, source-to-detector spacing, or some combination thereof. For the most accurate assays in cases in which the half-life of the transmission source isotope is short, a mathematical decay calculation to determine current source strength should be made for each measurement. In the case of assays where gamma-ray peaks from the transmission source interfere with determination of the area of the gamma-ray peak used for nuclide analysis, peak fitting software may be able to resolve overlapping peaks or a two-pass assay may be used. In cases that employ a two-pass assay, equip the transmission source collimator with a computer-actuated shutter, preferably tungsten, to block the transmission source from the gamma-ray detector during one of the passes (see Fig. 1). As a safety consideration, design such shutters so that, in the event of a power failure, the shutter will shut off the radiation beam automatically.

7.2.7 *Absorber Foils*—Foils must generally be used to reduce the contribution of low-energy gamma rays to the overall count rate, especially in the assay of $^{239}$Pu. As mentioned in 6.3, cadmium or tin foils serve to absorb the low-energy gamma rays from the item. For $^{239}$Pu assay, a series of 0.5-mm (approximately 0.020-in.) cadmium or tin foils can serve for sensitivity versus interference optimization. The use of lead foil is likely to require the additional use of cadmium or tin and copper foils as secondary absorbers (closest to the detector) to reduce the intensity of the fluorescent X rays produced in the lead foil. A single 1-mm cadmium or tin foil may be appropriate for $^{235}$U assay. Once a combination is chosen, it cannot be changed without instrument recalibration.

## 8. Calibration and Reference Materials

8.1 *Calibration*:

8.1.1 *Isotope Specific (Mass) Calibration* of a segmented gamma-ray scanning instrument involves using a series of calibration items to determine the relationship between the observed, totally corrected count rate of a nuclide's characteristic gamma ray and the mass of nuclide known to be present.

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For this method, the radioisotope calibrated for is actually present in the calibration standards. With the correction of individual segment count rates for rate-related losses and the attenuation of each segment, a direct proportionality between count rate, summed over all segments of an item, and total nuclide mass is obtained. Guide C1156 provides background information useful in developing a calibration plan. See 10.3.2 through 10.3.12, 10.3.14, and 10.3.15 for details.

8.1.2 *Efficiency Calibration* of a segmented gamma-ray scanning instrument involves using calibration standards to determine for each segment the ratio of the observed, corrected count rate of each of a number of gamma rays from the standard to its known emission rate within the segment, and use these ratios (efficiencies) to define the system detection efficiency as a function of energy. A detector efficiency value appropriate to the gamma ray energy is then applied to each individual segment corrected count rate (corrected for attenuation and rate-related losses) and summed over all segments of an item. The sum is adjusted by the specific activity and gamma ray intensity to determine the radionuclide mass. For this method, the radioisotopes in the calibration standards are chosen based on their half-lives and gamma energies and typically are not the same as the isotopes for which the calibration is used. See 10.3.2 through 10.3.11 and 10.3.13 through 10.3.15.

8.1.3 Perform calibrations using the same procedures and conditions that will be used for the assays of actual waste items. These include, but are not limited to, electronic components, peak area determination procedures, procedures for the determination of counting losses, segment sizes, absorber foil combinations, collimator arrangements, and measurement geometries. Alternatively, differences between calibration and assay geometries can be corrected for by appropriately calculated correction factors **(13)**.

8.1.4 Ref **(5)**, Guide C1128, and NRC Regulatory Guide 5.53 provide useful guidelines for the preparation and characterization of calibration materials and calibration procedures and the statistical analysis of data. Where there are conflicts among the documents, Ref **(5)** reflects information most specific to SGS requirements.

8.2 *Reference Materials for Isotope Specific Calibration*:

8.2.1 Prepare small item calibration items by uniformly dispersing known masses of stable chemical compounds with a known isotopic mass fraction of the radionuclide of interest throughout a stable diluting medium such as graphite, diatomaceous earth, or castable silicon compounds. The radioactive material should have a particle size small enough so that the effects of self-attenuation within each particle are negligible. With this requirement satisfied, choose the best particle size range to form a stable, homogenous mixture with the diluting material. Although the segmentation procedure used by the instrument usually compensates for stratification of the components of the mixture over time, some mixing, provided by gently shaking or rolling the container prior to each measurement, may be useful for calibration items containing powder.

8.2.2 Construct calibration items for larger item types such as 208-L (55-gal) drums from modules of matrix material such as filter paper, fiberglass, etc., wetted with known quantities of solutions containing the nuclide of interest at a known concentration. Dry the modules and pack them in plastic bags. Place the modules into the drum in a uniform manner until the drum is filled. Modules with varying nuclide loadings and varying combinations of modules produce a range of item loadings. For purposes of the initial calibration process, the mass of nuclide in individual modules should be limited so as not to create self-attenuating lumps (Note 1). Where possible, eliminate voids and small volumes containing high concentrations of nuclide **(14)**.

8.2.3 For each item geometry, prepare a set of three calibration items of differing nuclide mass. The mass loadings and the gamma-ray transmissions through the calibration items should span the ranges expected in the unknowns.

8.2.4 In order to evaluate the magnitude of biases that will be caused by the deviation of real items from ideal distributions of matrix and nuclide, prepare representative items from segregated varieties of scrap and waste materials typical of expected assay items. Vary the spatial distribution of the nuclide from widely dispersed to concentrated in various extreme dimensions of the container volume. Comparison of the assay results for such representative items with the known nuclide masses will indicate the possible range of bias caused by heterogeneity of nuclide and matrix material and that caused by nuclide location within the item.

8.2.5 Nuclide particle sizes in assay items may vary from those in the calibration standards, causing variations in the count rate per gram of nuclide and yielding biased results. An acceptable alternative to the preparation of special representative standards for calibration and uncertainty estimation measurements is the assay of real items (actual process materials) by analytical methods less sensitive to particle size problems (see NRC Regulatory Guide 5.53). These analytical methods may be total dissolution and solution quantification after completion of the segmented gamma-ray measurements **(15)**, or combined gamma-ray isotopic and calorimetric assay for plutonium materials. In either case, the determination of biases for these items will require special attention.

8.3 *Reference Materials for Efficiency Curve Calibration*:

8.3.1 Radionuclide sources for determining an efficiency curve are typically multi-isotope sources having multiple gamma ray energies spanning a broad energy range. The available gamma ray energies should be sufficient to appropriately define the efficiency function over the energy range of interest, generally 50 to 2000 keV.

8.3.2 Line sources prepared by radioisotope source vendors are often used. Line source uncertainties are generally in the range of a few percent at 1 standard deviation. Uncertainties in the data for radionuclide half-lives and gamma ray emission intensities also contribute to the measurement uncertainty. Each of these uncertainties must be included in an uncertainty propagation to determine the total measurement uncertainty