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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 C 1133; 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 nondestructive assay 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. 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 vertical segments of the container. Corrections are also made for counting losses occasioned by signal processing limitations **(1–3)**.[2]

1.2 There are currently at least three systems in use or under active development for determining the attenuation experienced by the radiation emitted from the nuclide of interest. These methods include the following: the original segmented gamma scan transmission procedure (SGS) **(4,5)**; a procedure involving measurements of the transmission at multiple energies combined with corrections for nuclide lumps based on assays of the nuclide of interest at multiple energies (MESGS) **(6–8)**; and tomographic scanning procedures (TGSS) **(9,10)**.

1.2.1 The simplest procedure, the original segmented gamma scan transmission procedure, will be covered in detail in the remainder of the main body of this test method and Annex A1.

1.2.2 Due to the limited experience and literature documenting the MESGS and TGS procedures, discussion in this test method will be limited to the above references.

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

1.3.1 The particles containing the nuclides of interest must be small to minimize self absorption of emitted gamma radiation.

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

1.4 The assay technique may be applicable to loadings of from one to several hundred grams of nuclide, with more restricted ranges to be applicable depending on specific packaging and counting equipment considerations. Measured transmission values must be available to permit valid attenuation corrections.

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

1.6 *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:*

C 982 Guide for Selecting Components for Energy-Dispersive X-Ray Fluorescence (XRF) Systems[3]

C 1128 Guide for Preparation of Working Reference Materials for Use in the Analysis of Nuclear Fuel Cycle Materials[3]

C 1156 Guide for Establishing Calibration for a Measurement Method Used to Analyze Nuclear Fuel Cycle Materials[3]

C 1207 Test Method for Nondestructive Assay of Plutonium in Scrap and Waste by Passive Neutron Coincidence Counting[3]

C 1210 Guide for Establishing a Measurement System Quality Control Program for Analytical Chemistry Laboratories within the Nuclear Industry[3]

E 181 Test Methods for Detector Calibration and Analysis of Radionuclides[4]

2.2 *ANSI Standards:*[5]

ANSI N15.20 Guide to Calibrating Nondestructive Assay Systems

ANSI N15.35 Guide to Preparing Calibration Material for Nondestructive Assay Systems that Count Passive Gamma Rays

[1] This test method is under the jurisdiction of ASTM Committee C-26 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] *Annual Book of ASTM Standards*, Vol 12.01.

[4] *Annual Book of ASTM Standards*, Vol 12.02.

[5] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

ANSI N15.37 Guide to the Automation of Nondestructive Assay Systems for Nuclear Materials Control
ANSI/IEEE 325 Test Procedures for Germanium Gamma-Ray Detectors
ANSI/IEEE 645 Test Procedures for High-Purity Germanium Detectors for Ionizing Radiation

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 a characteristic gamma ray from each nuclide. Corrections are made for count rate-related losses and attenuation by the item. Comparison to similarly corrected gamma-ray intensities, observed during the measurement of appropriate calibration materials, provides the relationship between observed gamma-ray intensity and nuclide content.

3.2 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).

3.3 Count rate-dependent losses from pulse pile-up and analyzer deadtime are monitored and corrected for by electronic modules and radioactive sources.

[6] Available from U.S. Nuclear Regulatory Commission, Public Document Room, 1717 H St., N.W., Washington, DC 20555.

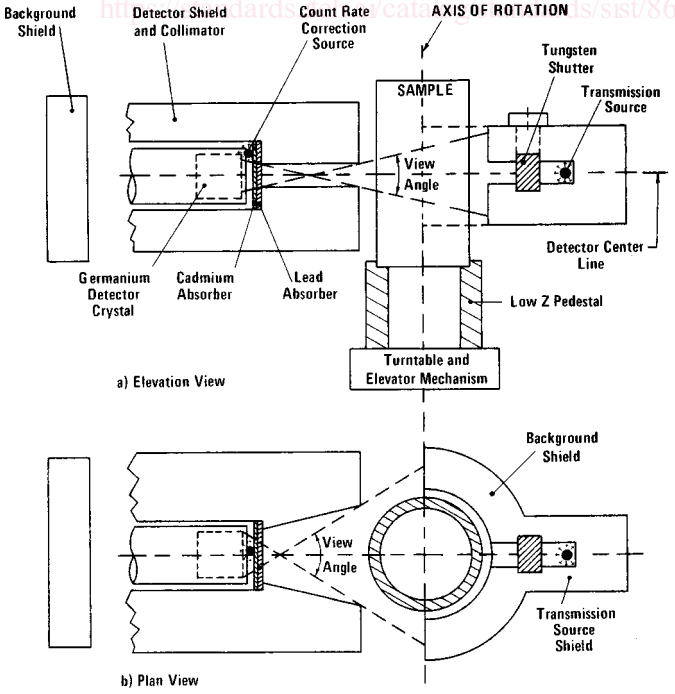

FIG. 1 General Arrangement for Segmented Gamma-Ray Scanning

3.4 The average linear attenuation coefficient of each vertical segment is calculated by measurement of the transmitted intensity of an external gamma-ray source. The source is mounted directly opposite the gamma-ray detector, on the far side of the assay item (see Fig. 1).

3.5 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, count rates are summed, and mass values for the nuclides of interest in the entire container are calculated based on comparisons to appropriate calibration materials. Based on counting statistics for individual segments, precision values are propagated to obtain the estimated precision of the analysis.

3.6 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.

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 The procedure is highly automated and requires little operator interaction.

4.4 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.

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 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 produced by ^{239}Pu gamma rays. 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, 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.

5.2 In the special 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. The problem can be avoided entirely by making 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.

5.3 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 ANSI N15.20, ANSI N15.35, and 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 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 should provide a sufficiently accurate determination of attenuation.

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

Nuclide	Peak Energy, keV	Transmission 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
^{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	^{75}Se	400.1	^{133}Ba	356.3
^{241}Am	662.4	^{75}Se	400.1	^{133}Ba	356.3

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 measurement item and the detector (see Fig. 1 and 7.2.15).

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 correction 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 measurement item itself, but not from the nuclide of interest, it may be possible to eliminate them from future measurement items by scrap and waste segregation procedures. Such procedures are discussed in detail in NRC Regulatory Guide 5.11.

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

6.6 Some matrix forms are inherently unsuitable for the original segmented gamma-ray analysis procedures.

6.6.1 Such forms may contain lumps of nuclide, that is, nuclide contained in small volumes of matrix material 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. Approximately 15 % of the 186-keV ^{235}U gamma rays will be absorbed in a uranium metal sphere of the same diameter.

6.6.2 The presence of lumps of plutonium may be detected and, in some cases, a corrected value calculated using the MESGS technology. The technique uses transmission-corrected assay results for multiple gamma-ray energies from a single isotope and a weighting function to account for self-absorption by lumps. This approach has been used only for the

analysis of ^{239}Pu using a ^{75}Se transmission source, where both the nuclide of interest and the transmission source emit 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.

6.6.3 Another condition that will cause measurement problems is presented by containers with several irregular regions, highly variable in density, 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, such as passive neutron coincidence counting as described in Test Method C 1207, should be used.

6.7 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 measurement item. Simple overscanning 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 overscanning 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 C 982 and NRC Regulatory Guide 5.9. Data acquisition systems are considered in ANSI N15.37 and 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 *High-Resolution, Coaxial Germanium Detector*—Detector equipped with a high-count rate, resistive feedback preamplifier. Coaxial detectors should have a relative efficiency of 10 % or greater (ratio of the area under the 1.33-MeV peak of ^{60}Co to that obtained with a 76 by 76 mm (3 by 3 in.) NaI(Tl) detector, at a source to detector distance of 25 cm). 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 E 181, ANSI/IEEE 325, and ANSI/IEEE 645.

7.2.2 *Spectroscopy Grade Nuclear-Pulse Amplifier*—Amplifier offering selectable pulse shaping time constants (1, 2, and 4 µs values should be available as a minimum), pole zero adjustment, active gated baseline restoration, pulse pile-up rejection, and a preamplifier power supply. The pulse pile-up rejection signals from the amplifier must be compatible with the multichannel analyzer described in 7.2.7. A discussion of these functions is given in Guide C 982.

7.2.3 *Oscilloscope*—Oscilloscope providing selectable time bases ranging from 1 ms/cm to 0.5 µs/cm (20 MHz) and selectable vertical sensitivities ranging from 5 V/cm to 10 mV/cm for proper adjustment of the various amplifier controls is required.

7.2.4 *Spectrum Stabilizer*—Stabilizer monitoring two separate spectrum peaks, to control changes in both energy gain and zero intercept. The stabilizer must be compatible with the multichannel analyzer described in 7.2.7.

7.2.5 *High-Voltage Bias Supply*—Supply equipped with a continuously adjustable voltage control and with a voltage range compatible with the requirements of the detector above.

7.2.6 *Count-Rate Meter*—Meter compatible with output provided by the amplifier.

7.2.7 *Multichannel Analyzer*—Analyzer with a minimum of 4096 data channels is recommended. The analyzer should operate using a Wilkinson-type analog-to-digital converter (ADC) with a minimum ADC clock rate of 100 MHz, or a fixed conversion time ADC with a maximum conversion time of 10 µs. Facilities must be provided to activate the analyzer functions by the controlling computer and to transmit count data to the computer. All of these functions may be provided in either a single unit or in two components, an ADC and a separate data storage unit. Facilities for spectrum display may be provided in either the analyzer itself or separately in equipment compatible with the ADC/data storage units.

7.2.8 *Computer*—Computer equipped with sufficient memory and disk mass storage is required for system control, data reduction, and report generation. Interface capability for computer control of analyzer functions, scan table control, operator input to the system, and analytical report output must also be provided.

7.2.9 *Interactive Terminal*—Terminal compatible with the computer described in 7.2.8 is required for system and measurement control.

7.2.10 *Hardcopy Printer*—Printer compatible with the computer described in 7.2.8 is required for system documentation and analytical report generation.

7.2.11 *Motorized, Vertical Scanning Turntable*—Turntable capable of accommodating the largest size and weight containers to be measured is required. Computer-actuated methods for controlling vertical movement include timers and stepping motors and allow a choice of segment size. For normal analyses, segment sizes between the height of the collimator and one-half the collimator height provide sufficient segmentation. Segment sizes equal to one-half the height of the collimator slit provide the maximum sensitivity to nuclide located in any portion of the container. Vertical movement

repeatability within ± 0.5 % of the segment height should be available. Both helical or fixed-segment counting schemes are acceptable. 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.

7.2.12 *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 measurement item, reduces the viewing angle and improves segmentation. The reduced viewing angle decreases the bias of the attenuation correction and decreases the severity of end effects. These benefits must be balanced against a decrease in overall sensitivity (count rate/gram), due both to the more restricted field of view and to the greater distance of the detector from the nuclide. Collimator slit heights should be chosen so as to be in the range of 1⁄10 to 1⁄15 of the height of the measurement item. The horizontal field of view must include the entire diameter of the item.

7.2.12.1 *Large Items*—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 is reasonable.

7.2.12.2 *Small Items*—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.

7.2.13 *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 to the cryostat of 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 on the cryostat is adjusted to produce a count rate providing sufficient precision for the assay times used and then fixed.

7.2.14 *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 mCi 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 slice of the measurement item. Table 1 provides a listing of suggested nuclides for use as transmission sources, with the listed nuclides of interest. Because some of the suggested source isotopes are relatively short-lived, it may be necessary to obtain them with an activity considerably above the optimum to provide for a useful working life. The count rate of new sources 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 required. In cases that require 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.

7.2.15 *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 7.3, 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 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 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. 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 C 1156 provides background information useful in developing a calibration plan. See 10.3.2 through 10.3.13 for details.

8.1.2 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.

8.1.3 Ref **(5)**, Guide C 1128, ANSI Standards N 15.20 and N15.35, 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*:

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 (see ANSI N15.35). 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 **(12)**.

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 measurement items may vary from those in the calibration items, causing variations in the count rate per gram of nuclide and yielding biased results. An acceptable alternative to the preparation of special representative items for calibration and uncertainty estimation measurements is the assay of real items by analytical methods less sensitive to particle size problems (see ANSI N15.35 and NRC Regulatory Guide 5.53). These analytical methods may be total dissolution and solution quantification after completion of the segmented gamma-ray measurements **(13)**, or combined gamma-ray isotopic and calorimetric assay for plutonium materials. In either case, the determination of biases for real items will require special attention.

9. Precautions

9.1 *Safety*:

9.1.1 Transuranic materials are both radioactive and toxic. Adequate laboratory facilities and safe operating procedures must be considered to protect operators from both unnecessary exposure to ionizing radiation and contamination while handling measurement items **(11)**.

9.1.2 The recommended analytical procedures call for the use of radioactive isotope sources, some with high levels of ionizing radiation. Consult a qualified health physicist or radiation safety professional concerning exposure problems and leak test requirements before handling discrete radioactive sources.

9.2 *Technical*:

9.2.1 Prevent counting conditions that may produce spectral distortions. Use pulse pile-up rejection techniques if high count rates are encountered. Use absorbers when appropriate, to reduce the intensity of low-energy gamma rays such as the 59.54-keV emission of ^{241}Am (see 6.3 and 7.2.15). Temperature and humidity fluctuations in the measurement environment may cause gain and zero-level shifts in the gamma-ray spectrum. Use environmental controls or digital stabilization to prevent shifts, or use software to monitor the changes in gain and zero level, and adjust the regions of interest accordingly. Failure to isolate electronic components from other electrical equipment or the presence of noise in the ac power also may produce spectral distortions.

9.2.2 Locate the instrument in an area with as low a gamma-ray radiation background as possible. Prohibit the movement of containers of radioactive material in the vicinity of the instrument while an assay is underway.

10. Procedure

10.1 *Optimization of System Physical Parameters*:

10.1.1 Adjust the instrument controls to optimize signal processing and peak analysis functions. Choose the shaping time constant to optimize the trade-off between improved resolution with longer time constants and decreased dead time losses with shorter time constants. Time constants of 1 to 4 μs are commonly used. Choose the system gain so that a sufficient number of channels will be included in peaks to allow visual inspection of peak shapes, without including so many channels that peaks do not develop into recognizable shapes with expected count rates in planned count times. Generally peak shapes can be evaluated by including 10 to 20 channels between the one-tenth maximum boundaries of the peaks. Adjust pole zero and baseline restorer controls, using an oscilloscope in accordance with the manufacturer's instructions. Regions of interest around peaks to be used for analysis may be set manually by the operator or semiautomatically by the computer or analyzer, depending on the software package used.

10.1.2 Choose collimator sizes that are appropriate to the item type to be assayed, using the criteria described in 7.2.12.

10.1.3 Choose scanning segment sizes that match the item and previously chosen collimator sizes. For normal analyses, when stepped segments are used, limit the segment sizes to between the height of the collimator slit and one-half the height of the collimator slit. When helically scanned segments are used, segments considerably larger than the collimator slit height may be used.

10.1.4 Choose absorber combinations for the detector that