Designation: C1718 – 10

Standard Test Method for Nondestructive Assay of Radioactive Material by Tomographic Gamma Scanning[1]

This standard is issued under the fixed designation C1718; 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 describes the nondestructive assay (NDA) of gamma ray emitting radionuclides inside containers using tomographic gamma scanning (TGS). High resolution gamma ray spectroscopy is used to detect and quantify the radionuclides of interest. The attenuation of an external gamma ray transmission source is used to correct the measurement of the emission gamma rays from radionuclides to arrive at a quantitative determination of the radionuclides present in the item.

1.2 The TGS technique covered by the test method may be used to assay scrap or waste material in cans or drums in the 1 to 500 litre volume range. Other items may be assayed as well.

1.3 The test method will cover two implementations of the TGS procedure: *(1)* Isotope Specific Calibration that uses standards of known radionuclide masses (or activities) to determine system response in a mass (or activity) versus corrected count rate calibration, that applies to only those specific radionuclides for which it is calibrated, and *(2)* Response Curve Calibration that uses gamma ray standards to determine system response as a function of gamma ray energy and thereby establishes calibration for all gamma emitting radionuclides of interest.

1.4 This test method will also include a technique to extend the range of calibration above and below the extremes of the measured calibration data.

1.5 The assay technique covered by the test method is applicable to a wide range of item sizes, and for a wide range of matrix attenuation. The matrix attenuation is a function of the matrix composition, photon energy, and the matrix density. The matrix types that can be assayed range from light combustibles to cemented sludge or concrete. It is particularly well suited for items that have heterogeneous matrix material and non-uniform radioisotope distributions. Measured transmission values should be available to permit valid attenuation corrections, but are not needed for all volume elements in the container, for example, if interpolation is justified.

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

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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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

C1490 Guide for the Selection, Training and Qualification of Nondestructive Assay (NDA) Personnel

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

C1592 Guide for Nondestructive Assay Measurements

C1673 Terminology of C26.10 Nondestructive Assay Methods

2.2 *ANSI Standards:*[3]

ANSI N15.37 Guide to the Automation of Nondestructive Assay Systems for Nuclear Materials Control

2.3 *Nuclear Regulatory Commission (NRC) Guides*[4]

NRC Guide 5.9 Guidelines for Germanium Spectroscopy Systems for Measurement of Special Nuclear Material, Revision 2, December 1983

[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.

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[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.

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

[4] Available from U.S. Nuclear Regulatory Commission, Washington, DC 20555-0001, http://nrc.gov.

NRC Guide 5.53 Qualification, Calibration, and Error Estimation Methods for Nondestructive Assay, Revision 1, February 1984

3. Terminology

3.1 *Definitions:*

3.1.1 Terms shall be defined in accordance with Terminology C1673 except for the following:

3.1.2 *Algebraic Reconstruction Technique (ART), n*—image reconstruction technique typically used in the TGS method to obtain the transmission map as a function of atomic number (Z) and gamma ray energy (**1**).[5]

3.1.3 *aperture, n*—the terminology applies to the width of the detector collimator. In the case of a diamond collimator, the aperture is defined as the distance between the parallel sides of the diamond. In some designs, the detector collimator can be a truncated diamond that consists of flat trim pieces at the left and right corners of the diamond. This type of collimator is usually designed with the distance between the trim pieces set equal to the distance between the parallel surfaces (aperture).

3.1.4 *voxel, n*—volume element; the three-dimensional analog of a two-dimensional pixel. Typically 5 cm on a side for a 208 L drum.

3.1.4.1 *Discussion*—The full container volume will be divided into a number of smaller volume elements (typically 100–2000 or typically 0.1 % of the total container volume), which are not necessarily rectilinear.

3.1.5 *Beers Law, n*—the law states that the fraction of uncollided gamma rays transmitted through layers of equal thickness of an absorber is a constant. Mathematically, Beer's Law can be expressed as follows:

$$T = \frac{I}{I_0} = exp\left\{ -\frac{\mu}{\rho}\cdot\rho\cdot t \right\}$$

In the above equation, I_0 is the intensity of a pencil beam of gamma rays incident on a uniform layer of absorber, I is the transmitted intensity through the layer, μ/ρ is the mass attenuation coefficient of the absorber material, ρ is the density of the absorber and t is the thickness of the layer. For a heterogeneous material the exponent would be integrated along the ray path.

3.1.6 *expectation maximization (EM), n*—image reconstruction technique typically used in the TGS method to solve for the emission map as a function of gamma ray energy (**2, 3**).

3.1.7 *grab (or view), n*—a single measurement of the scan, where the scan sequence consists of measurements at various heights, rotational positions, and translation positions of the assay item.

3.1.8 *map (transmission and emission), n*—a voxel by voxel record of the matrix density or linear attenuation coefficient (transmission map) or a voxel by voxel record of radionuclide content (emission map).

3.1.9 *material basis set (or MBS), n*—the method where the linear attenuation coefficient map for a matrix material is determined in terms of 2 or 3 basis elements that span the Z range of interest (**4**).

3.1.10 *non-negative least squares (NNLS), n*—constrained least squares fitting algorithm used in TGS analysis to obtain an initial estimate of the transmission map.

3.1.11 *pre-scan, n*—a preliminary scan of an assay item employed by some TGS implementations to optimize the scan protocol on an item-by-item basis.

3.1.12 *scan, n*—sequence of measurements at various heights, rotational positions, and translation positions of the assay item.

3.1.13 *response function, n*—detector efficiency (absolute or relative) as a function of measurement locus and gamma ray energy.

3.1.14 *tomography, n*—the mathematical method in which gamma ray measurements are used to determine the attenuation and emission characteristics of an item on a voxel-by-voxel basis.

3.1.15 *translation, n*—the relative motion in the horizontal direction of the item to be measured perpendicular to the transmission source-detector axis.

3.1.16 *TGS Number, n*—uncalibrated result of a TGS analysis representing count rate corrected for geometrical efficiency, gamma ray attenuation , and rate loss at a given emission gamma ray energy, proportional to the mass or activity of a specific radionuclide.

3.1.17 *view, n*—see *grab*.

4. Summary of Test Method

4.1 Assay of the radionuclides of interest is accomplished by measuring the intensity of one or more characteristic gamma rays from each radionuclide utilizing TGS techniques. TGS techniques include translating, rotating and vertically scanning the assay item such that a 3-dimensional (3D) image can be reconstructed from the data. Generally two 3D images are constructed; a transmission image and a passive emission image. Corrections are made for count rate-related losses and attenuation by the matrix in which the nuclear material is dispersed. The calibration then provides the relationship between observed gamma ray intensity and radionuclide content.

4.2 Calibration is performed using standards containing the radionuclides to be assayed or using a mixture of radionuclides emitting gamma rays that span the energy range of interest. The activities or masses of the radionuclides and the gamma ray yields are traceable to a national measurement database.

4.2.1 Using a traceable mixed gamma ray standard that spans the energy range of interest will enable the determination of the TGS calibration parameters at any gamma ray energy of interest, not just those that are present in the calibration standard. A calibration curve is generated that parameterizes the variation of the TGS calibration factor as a function of gamma ray energy.

4.3 The assay item is rotated about its vertical axis. Concurrently, the relative position of the assay item and detector are translated. This is repeated for every vertical segment. During this process, a series of measurements (grabs) are taken of gamma rays corresponding to the transmission source and the emission sources. A transmission scan is

[5] The boldface numbers in parentheses refer to a list of references at the end of this standard.

performed with the transmission source exposed. A separate emission scan is performed with the transmission source shielded.

4.3.1 From the transmission measurements, a 3D map of the average linear attenuation coefficient across of each voxel is determined.

4.3.2 From the emission measurements, a 3D map of the location of the gamma emitting radionuclides is determined. These 3D maps are typically low spatial resolution (for example, approximately 1/10 th the diameter would be a typical characteristic dimension).

4.3.3 Through a voxel by voxel application of Beer's Law, the emission source strength is corrected for the attenuation of the matrix material.

4.4 Count rate-dependent losses from pulse pile-up and analyzer deadtime are monitored and corrected.

4.5 The TGS determines an estimate of the average attenuation coefficient of each voxel in a layer of matrix using an over determined set of transmission measurements.

4.6 A collimator is used in front of the detector to restrict the measurement to a well-defined solid angle.

4.7 The TGS technique assumes the following item characteristics:

4.7.1 The particles containing the radionuclides of interest are small enough to minimize self-absorption of emitted gamma radiation. Corrections to slef-attenuation may be applied post TGS analysis, but is outside the scope of this standard.

4.7.2 The mixture of material within each item voxel is sufficiently uniform that an attenuation correction factor, computed from a measurement of gamma ray transmission through the voxel, is appropriate.

4.8 Typically, a single isotope of an element is measured, therefore when the total element mass is required, it is necessary to apply a known or estimated radionuclide/total ratio to the radionuclide assay value to determine the total element content (see Test Method C1030).

5. Significance and Use

5.1 The TGS provides a nondestructive means of mapping the attenuation characteristics and the distribution of the radionuclide content of items on a voxel by voxel basis. Typically in a TGS analysis a vertical layer (or segment) of an item will be divided into a number of voxels. By comparison, a segmented gamma scanner (SGS) can determine matrix attenuation and radionuclide concentrations only on a segment by segment basis.

5.2 It has been successfully used to quantify ^{238}Pu, ^{239}Pu, and ^{235}U. SNM loadings from 0.5 g to 200 g of ^{239}Pu (**5, 6**), from 1 g to 25 g of ^{235}U (**7**), and from 0.1 to 1 g of ^{238}Pu have been successfully measured. The TGS technique has also been applied to assaying radioactive waste generated by nuclear power plants (NPP). Radioactive waste from NPP is dominated by activation products (for example, ^{54}Mn, ^{58}Co, ^{60}Co, ^{110m}Ag) and fission products (for example, ^{137}Cs, ^{134}Cs). The radionuclide activities measured in NPP waste is in the range from 3.7E+04 Bq to 1.0E+07 Bq. Some results of TGS application to non-SNM radionuclides can be found in the literature (**8**).

5.3 The TGS technique is well suited for assaying items that have heterogeneous matrices and that contain a non-uniform radionuclide distribution.

5.4 Since the analysis results are obtained on a voxel by voxel basis, the TGS technique can in many situations yield more accurate results when compared to other gamma ray techniques such as SGS.

5.5 In determining the radionuclide distribution inside an item, the TGS analysis explicitly takes into account the cross talk between various vertical layers of the item.

5.6 The TGS analysis technique uses a material basis set method that does not require the user to select a mass attenuation curve apriori, provided the transmission source has at least 2 gamma lines that span the energy range of interest.

5.7 A commercially available TGS system consists of building blocks that can easily be configured to operate the system in the SGS mode or in a far-field geometry.

5.8 The TGS provides 3-dimensional maps of gamma ray attenuation and radionuclide concentration within an item that can be used as a diagnostic tool.

5.9 Item preparation is limited to avoiding large quantities of heavily attenuating materials (such as lead shielding) in order to allow sufficient transmission through the container and the matrix.

6. Interferences

6.1 Radionuclides may be present in an item that produce gamma rays with energies the same as or very nearly equal to the gamma rays of the radionuclide to be measured or of the transmission source. There may be instances where emission gamma rays from multiple radionuclides interfere with one another or with a gamma ray present in the background. A few examples are given below:

6.1.1 *Interference with Transmission Gamma Rays:*

6.1.1.1 In TGS systems where an ^{152}Eu source is used as the transmission source, one has to consider the following interferences while assaying plutonium containing waste drums. *(1)* Transmission data from the 121.78 keV gamma ray from ^{152}Eu may be affected by Pu K-Xrays. The interference can be corrected by subtracting the emission background from the transmission spectra on a view by view basis. *(2)* Transmission data from the 411.2 keV gamma ray from ^{152}Eu may be affected by the 413.7 keV gamma ray peak from ^{239}Pu. In such cases, the 411.2 keV can be used to calculate transmission only if the emission background has been subtracted. *(3)* Transmission data from the 344.28 keV gamma ray from ^{152}Eu may be affected by the 345.01 keV gamma ray peak from ^{239}Pu. However, the 344.28 keV peak from ^{152}Eu has a relatively high yield and the interference from the ^{239}Pu gamma ray may be negligible. Subtracting the emission background on a view by view basis will eliminate the bias.

6.1.1.2 In the special case of single pass assays (emission and transmission data collected together) of ^{239}Pu waste 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 could interfere with the 413.7 keV ^{239}Pu peak. 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, one pass with the transmission shutter open and another pass with the shutter closed.

6.1.2 *Interference among Emission Gamma Rays:*

6.1.2.1 In waste items containing ^{137}Cs and ^{241}Am, the 661.6 keV gamma ray from ^{137} Cs and the 662.4 keV gamma ray from ^{241}Am can interfere with each other. The 721.9 keV gamma ray of ^{241} Am may be useful as an alternative as well as for extracting the 662.4 keV peak area based on branching ratios and detector response. Thereafter, the 661.6 keV peak from ^{137}Cs can be corrected for interference.

6.1.2.2 The 415.8 keV gamma ray from the daughter decay of ^{237}Np can interfere with the 413.7 keV gamma ray of ^{239}Pu. In addition, there are several other gamma rays in the 300–400 keV region. Peaks from these gamma rays could interfere with the 413.7 keV ^{239}Pu peak and several other often-used peaks produced by ^{239}Pu gamma rays. The 129.3 keV gamma ray may be used as a reasonable alternative, if attenuation at this energy will not preclude analysis or substantially decrease precision due to poor counting statistics.

6.1.3 *Interference from Ambient Background:*

6.1.3.1 Peaks may appear at the gamma ray energies used for analysis when there is no item present on the rotating/translating platform. The likely cause is excessive amounts of radioactive sources or waste containers stored in the vicinity of the detector. The preferred solution to this problem is removal of the radioactive sources from the vicinity and restraining the movement of sources close to the system during measurements. If these conditions cannot be met, shielding must be provided to sufficiently eliminate these peaks. Shielding opposite the detector, on the far side of the item to be assayed, will also help reduce the amount of ambient radiation seen by the detector. The ambient background measurement must be taken (following the normal TGS assay protocol) with an item with a representative non-radioactive matrix loaded on to the turntable.

6.1.4 The background contributions can be subtracted during the TGS analysis. The emission background can be subtracted from transmission data, and the ambient background can be subtracted from the emission data. The two types of background subtractions are performed on a view by view basis.

7. Apparatus

7.1 In Fig. 1, the detector assembly is on the right hand side and the transmission assembly is to the left. The translating (and rotating) platform with the item loaded on it is shown in the middle. General guidelines for the selection of detectors and signal processing electronics are discussed in relevant operations manuals and NRC Guide 5.9. Data acquisition systems are considered in ANSI N15.37 and NRC Guide 5.9.

7.2 Complete hardware and software systems for TGS, of both large and small items, are commercially available. The specification and procurement of the hardware and software should follow a careful evaluation of the measurement quality objectives, expected materials to be assayed, and associated system costs. This evaluation should be completed by an NDA

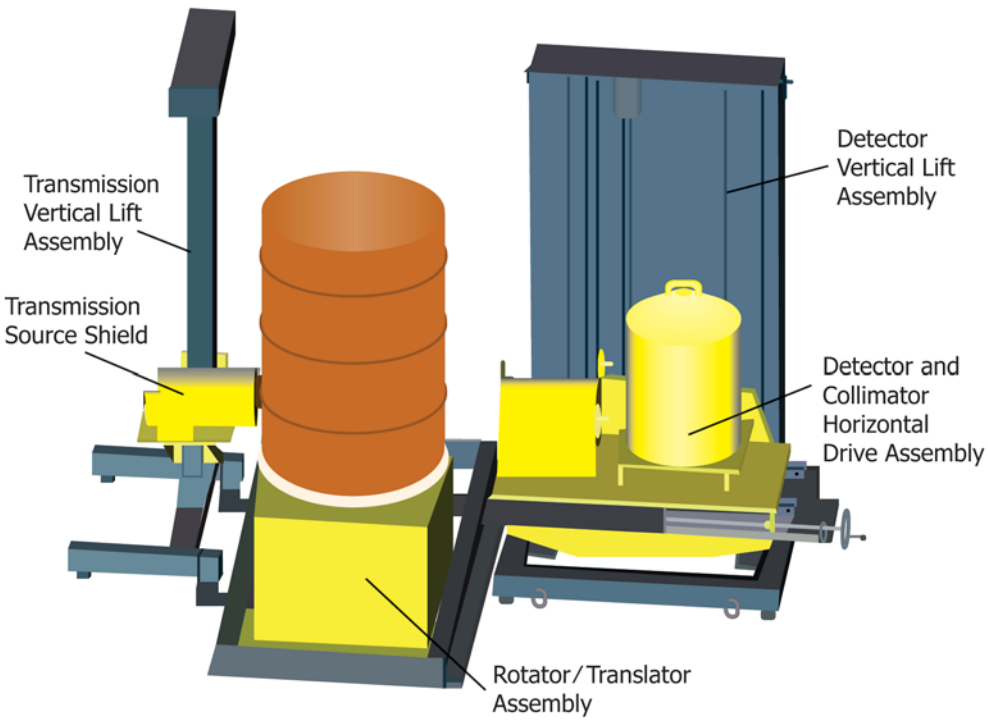

FIG. 1 Example of a Tomographic Gamma Scanning System

professional (Guide C1490). The system should have the following components:

7.2.1 *High-resolution, high purity germanium detector*—Detector resolution and efficiency shall be appropriate for the users specific application and needs as determined by an NDA professional (Guide C1490).

7.2.2 *Detector collimator*—The detector collimator opening shall be a reasonable compromise between spatial resolution and counting statistics, judged against the measurement objective. The count rate per grab of the TGS can be improved by using a wider collimator or a higher efficiency detector.

7.2.3 *External source of gamma rays from a transmission source*—An external source shall be used to interrogate the item and characterize the attenuation properties of matrix. (See Table 1 for suggested sources). The count rate per grab of transmitted gamma rays can be improved by using a transmission source of higher intensity.

7.2.4 *Motorized scanning system*—the items shall be scanned over three axes of motion relative to the detector (usually vertical translation, horizontal translation, and rotation about a vertical axis).

7.2.5 *Tomographic reconstruction algorithms*—TGS reconstruction algorithms shall be employed to determine a three-dimensional map of matrix density and radionuclide distribution.

TABLE 1 Commonly Used Transmission Source and Assay Radionuclide Combinations

Radionuclide of Interest	Peak Energy (keV)	Transmission Source	Peak Energy (keV)
^{235}U	185.7	^{169}Yb	177.2 198.0
^{238}Pu	152.7 766.4	^{75}Se	136.0 400.1
^{238}Pu	152.7 766.4	^{152}Eu	121.8 244.7 344.3 411.1 778.9
^{239}Pu	129.3 203.6 345.0 375.1 413.7	^{75}Se	121.1 136.0 264.7 279.5 400.1
^{239}Pu	129.3 203.6 345.0 375.1 413.7	^{152}Eu	121.8 244.7 344.3 411.1
^{239}Pu	129.3	^{57}Co	122.1 136.5
^{137}Cs	661.6	^{152}Eu	411.1 778.9
^{54}Mn	834.8	^{152}Eu	778.9 867.4 964.1
^{60}Co	1173.2 1332.5	^{152}Eu	964.1 1112.1 1408.0

7.3 *Rate-Loss Correction Source or a Pulser*—A ^{109}Cd source is commonly used as the reference source for performing rate loss corrections. Alternatively, a high precision pulser may be used for the same purpose. When a pulser is used, care needs to be taken in the set-up to avoid spectral distortion.

7.4 *Software*—The system should include one or more software tools for the collection of data, motion control of the system, and analysis of data. The system may include tools for performing isotopic data collection and analysis.

7.5 In two-pass assays, transmission gamma rays can be significantly attenuated by using a shutter made out of a high Z material.

7.6 To attenuate the X-rays from high Z collimator and shield material, the inner walls of the collimator and shield as well as the front face of the detector may be lined with a "graded shield" made of a layer of Sn and a layer of Cu.

8. Preparation of Apparatus

8.1 Perform calibrations using the same procedures and conditions that will be used for the assays of actual items. These include, but are not limited to, electronic components, peak area determination procedures, procedures for the determination of counting losses, voxel sizes, absorber foil combinations, collimator arrangements, and measurement geometries. Changing conditions will change the calibrations. Some commercial systems may allow certain parameters to change (for example, aperture, distance from item surface to detector, etc.) and allow the corresponding calibration factors to be selected.

8.2 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 4 to 8 μs are commonly used for analog pulse processing electronics. If a digital signal processor is used, select filter settings equivalent to the above-mentioned analog shaping times. Follow the manufacturer's instructions for setting time constants or filter settings.

8.3 Set the conversion gain on the analog-to-digital converter (ADC). Adjust the amplifier gain. Perform pole-zero cancellation (if a resistive feed-back pre-amplifier is used). Set up a restore rejection veto (reset inhibit) if a transistor reset pre-amplifier is used. Perform an energy and shape calibration of the detector. If a pulser is used for performing rate loss corrections, ensure that the amplitude and frequency of the pulses are set to the appropriate values. A significant advantage in using a pulser as opposed to a rate loss source is that the pulser peak can be placed at an energy where it will not interfere with the gamma ray peaks of interest.

8.4 *Pile-up at high rates*—Pulse pile-up can distort peak shapes and can bias the counts registered in the regions of interest (ROI) in the gamma ray spectra. The TGS technique relies on the counts in the ROIs to determine the transmission and emission maps. It is important to eliminate pulse pile-up. Pile-up rejection circuitry in the amplifier should be enabled to do this.

8.5 Set up the data acquisition and analysis software. Typically, the data acquisition software will interface with mechanism control hardware (stepper motors, DC motors, etc.) in order to ensure that the item is scanned properly. Additionally, the data acquisition software may also have the capability to automatically set an appropriate assay geometry (detector horizontal position, detector collimator aperture, etc.) based on drum dose rate or dead time. In such cases, the parameters for the assay geometry must be entered into the control software. The acquisition software also interfaces with the pulse processing electronics and the system computer to acquire data for a preset time, and store the data.

8.6 Choose collimator sizes that are appropriate to the item type to be assayed.

8.6.1 Collimator aperture must be selected based on *(1)* the distance of the container from the detector, *(2)* the count rate level (or surface dose rate of the container), *(3)* scanning diameter of the assay, and *(4)* the desired voxel grid.

8.6.2 The farther the detector is with respect to the container, the narrower the collimator aperture should be. For TGS systems used in industrial facilities, for a 208 litre drum where the outer surface is at a distance of 500 mm from the detector, a collimator aperture of 60 mm would be typical. If a 208 litre drum is at a distance of 1000 mm from the detector, a collimator aperture of 40 mm would be typical. For TGS systems used in a research facility, for assaying 208 litre drums, the distance from the surface of the drum to the detector is typically 200 mm.

8.6.3 The higher the surface dose rate of the container, the farther the detector should be, and narrower the collimator aperture. This should be done to maintain the spatial resolution, as well as to remain below the upper limit of the dynamic count rate range of the detector.

8.6.4 The collimator aperture is typically set 1 to 1.5 times the length of the voxel, based on sensitivity and precision in a given acquisition time.

8.7 Set up ROIs around gamma ray peak energies of interest for emission as well as transmission scans. For each peak, set up ROIs to cover the peak region and the continuum regions to the left and right of the peak. ROIs around peaks to be used for analysis may be set manually by the operator or semi-automatically by the computer or analyzer, depending on the software package used.

8.8 Set up the number of vertical layers over which the item will be scanned. For a 208 litre or a 300 litre drum, the number of vertical layers to be scanned is normally 16.

8.9 Set up acquisition and analysis software to perform the desired number of data acquisition grabs per scan and the assay time per scan. Also set up the software to analyze the data over the desired voxel grid.

8.10 Typically for a 208 litre drum, for a nominal 1h assay period, about 112 seconds are spent acquiring data at each of the 16 layers in each of the two modes (transmission and emission). Each layer is broken into a 10 × 10 lattice of square voxels (Fig. 2). By convention, based on signal-to-noise and robustness of the analysis arguments, the number of data grabs is set at 1.5 times the number of voxels (that is, roughly π/2 times the number of voxels that fit around the drum perimeter). Therefore for each of the 16 vertical layers, 150 measurements

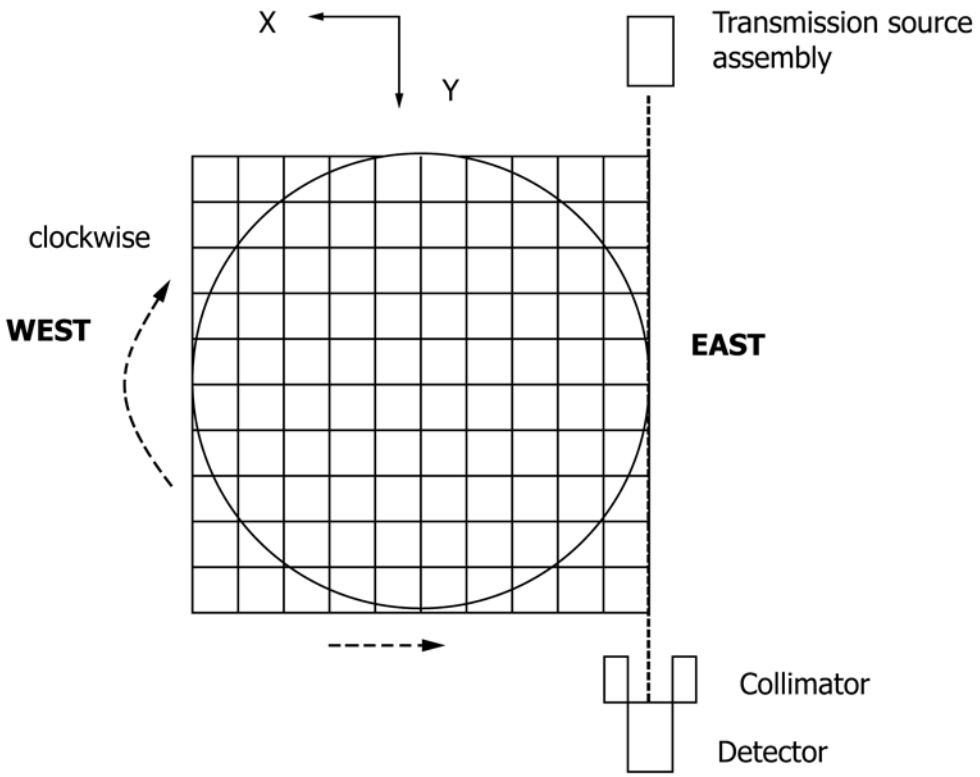

FIG. 2 Example of a TGS Voxel Grid Pattern

are made in order to mathematically over determine the solution for 88 voxels in the 10 × 10 grid in each layer (assuming all data grabs are valid).

8.10.1 Count time for each view (or grab), should be set based on considerations of counting precision and the overall assay time for the measurement requirement.

8.10.2 The number of views per scan per layer must be greater than the number of voxels in the grid per layer (typically 1.5 times greater, based on sampling theory).

9. Calibration and Reference Materials

9.1 Calibration of a TGS system relies on measurements of well-characterized reference materials containing known amounts of appropriate radionuclides. The radionuclide sources used are calibration standards whose activities or masses are traceable to a national measurements database. The calibration standards are distributed within a container with a well-characterized matrix. Such a configuration is called a reference material in this document. A TGS system calibrated using reference materials can be used to quantify radionuclides in items. A facility may use a "working reference" to calibrate the system if the objective is to track the relative performance of the TGS system for quality assurance purposes. A facility can create a working reference by distributing radionuclide sources, that are not calibration standards, inside a representative container matrix. A TGS system can be calibrated using calibration standards that contain: *(1)* only those radionuclides that are of interest in the item assays (isotope specific calibration), *(2)* radionuclides that are not necessarily of interest in the item assays but consist of gamma lines spanning the energy range of interest, and *(3)* a mixture of radionuclides that are of interest in item assays as well as those that are not expected in item assays. Calibration standards can consist of SNM radionuclides only, non-SNM radionuclides only, or a mixture of SNM and non-SNM radionuclides. Guides C1156 and C1592 provide additional information useful in developing and executing a calibration plan.

9.2 *Calibration:*

9.2.1 Calibration of a TGS instrument uses a series of reference materials to determine the relationship between the corrected count rate of a radionuclide's characteristic gamma ray and the mass or activity of radionuclide known to be present. After the correction of individual voxel count rates for rate-related losses and the attenuation of each voxel, a direct proportionality between count rate, summed over all voxels of an item, and total radionuclide mass or activity is determined.

9.2.2 An output of a TGS analysis is a quantity known as the "TGS number" and the uncertainty associated with it. The TGS number and its uncertainty are determined at each emission energy, and represent values proportional to the activity or mass of an assayed radionuclide inside the drum. During calibration, TGS assays are performed using reference materials and the TGS numbers are obtained as a function of gamma ray energy. The TGS calibration parameter at each energy of interest is simply the TGS number per unit activity (or mass).

9.2.3 A separate calibration must be performed for each geometry of interest (collimator aperture, distance of detector from the surface of the container).

9.2.4 After obtaining the calibration parameters, a series of verification measurements must be performed using reference materials to validate the calibration. The verification measurements must span the various geometries of interest, the range of activity or mass loadings of the radionuclides, the dynamic range of the expected matrix attenuations and different source distributions.

9.2.5 Repeat measurements (at least 6) of a given reference material must also be performed to establish the reproducibility of the TGS results.

9.2.6 An item assay that uses an isotope-specific calibration will yield masses or activities for those radionuclides that are the same as the ones used during calibration. The TGS number obtained from the analysis of the item drum is simply divided by the calibration factor at the corresponding gamma ray energy to obtain the radionuclide mass or activity.

9.2.7 If calibration standards with isotopes of interest are not available, a multi-isotope calibration standard that emits gamma rays spanning the energy range of interest can be used. When the gamma ray yields are factored in, the TGS calibration factor can be expressed in units of TGS number per gammas per second. The shape of the curve describing TGS no./gammas/sec as a function of energy is very similar to the intrinsic efficiency curve of the detector. By fitting a calibration curve to the TGS no./gammas/sec data points, it is possible to determine by interpolation the activity or masses of radionuclides that are not present in the calibration standard. Further, the similarity of the TGS calibration curve and the intrinsic efficiency curve can be exploited in extending the TGS calibration to energies beyond the lowest and highest gamma ray energy calibration data points. This extrapolation is done by determining a scaling factor based on relative efficiencies for a simple source-detector geometry.

$$ScaleFactor = \frac{\varepsilon(E > E_{max})}{\varepsilon(E_{max})} \quad (1)$$

$$(TGS\ No./gammas/sec)_{E>E_{max}} = Scale\_Factor \times (TGS\ No./gammas/sec)E_{max} \quad (2)$$

$$ScaleFactor = \frac{\varepsilon(E < E_{min})}{\varepsilon(E_{min})} \quad (3)$$

$$(TGS\ No./gammas/sec)_{E>E_{min}} = Scale\_Factor \times (TGS\ No./gammas/sec)E_{min} \quad (4)$$

Caution must be used in extending the TGS calibration beyond the range of calibration data. The hardware and software set up, the data acquisition and analysis steps, and the assay protocol are the same for the isotope-specific and non-isotope-specific calibrations. A major difference between the two methods with regard to the set up is the ROI set up for the emission scan. In the efficiency calibration method, emission ROIs must be set up for all the gamma ray peaks of interest, not just the ones associated with radionuclides in the calibration standard.

9.2.8 *Discussion of empty drum calibration and matrix drum calibration*—Guides C1128, C1592, and NRC Guide 5.53 provide useful guidelines for the preparation and characterization of reference materials and calibration procedures and the statistical analysis of data.

9.2.9 If a new geometry is needed, for example, for a special investigation, for which a direct calibration has not been performed, a subject matter expert may be able to apply mathematical tools to estimate the relative change in response and quantify the additional uncertainty.

9.3 *Reference Materials for an Isotope-Specific Calibration*—The suggestions given in Sections 9.2.1 through 9.2.4 are consistent with good practices in performing nondestructive assay measurements. If these recommendations cannot be followed because of practical difficulties, then appropriate uncertainty estimates must be determined and assigned to account for the differences between the reference material and the real items being assayed.

9.3.1 For TGS assay of small items, reference materials can be prepared 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, or the same as the items to be assayed, or are known so a correction can be applied. Although the mapping procedure used by the instrument usually compensates for stratification of the components of the mixture over time, some re-mixing, provided by gently shaking or rolling the container prior to each measurement, may be useful for calibration standards containing powder.

9.3.2 In order to evaluate the magnitude of biases that will be caused by the deviation of real items from ideal distributions of matrix and radionuclide, prepare representative items from segregated varieties of scrap and waste materials typical of expected assay items. Vary the spatial distribution of the radionuclide 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 radionuclide masses will indicate the possible range of bias caused by heterogeneity of radionuclide and matrix material and that caused by radionuclide location within the item.

9.3.3 Radionuclide particle sizes in assay items may vary from those in the calibration standards, causing variations in the count rate per g of radionuclide 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 Guide 5.53). These analytical methods may be total dissolution and solution quantification after completion of the tomographic gamma ray measurements, 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.

9.4 *Reference Materials for a Non-Isotope-Specific Calibration:*

9.4.1 Radionuclide sources for determining a calibration 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 for nuclear power plant waste assays, 50 keV to 1000 keV for waste containing SNM).

9.4.2 Line sources inserted into holes drilled at specific radial locations of a cylindrical container with a non-radioactive matrix material are commonly used (**9**). Line source uncertainties are generally in the range of a few percent at 1σ uncertainty level. 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 (TMU) of an instrument. The TMU should be determined for each container and material type.

10. Hazards

10.1 *Safety Hazards:*

10.1.1 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.

10.1.2 A TGS system uses a transmission source whose activity is typically 5 millicuries to 250 millicuries. The transmission source must be adequately shielded to avoid excessive exposure relative to facility-specific objectives.

10.1.3 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 assay items.

10.1.4 The recommended analytical procedures call for the use of radionuclide 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.

10.1.5 The TGS system consists of moving mechanical parts. Necessary safety precautions such as lights and alarm sounds must be used to indicate motion and commencement of motion. The system must be equipped with emergency stop buttons or switches that can be manually or automatically activated if a dangerous situation is encountered. Additionally, equipment such as overhead cranes or forklifts may have to be used to load and unload heavy containers. Care must be taken while operating these so that injury to personnel or damage to the system can be prevented.

10.2 *Technical Hazards:*

10.2.1 The mechanical movement (translation and rotation) of the platform must be synchronized and maintained at a constant rate. If the rate of motion changes during data acquisition, the image re-construction will be severely affected and will bias the TGS results. Routine maintenance must be performed to keep the mechanical parts and the stepper motors in good working condition.

10.2.2 The TGS method requires that ROIs be defined around the gamma ray energies of interest. A peak ROI and a background ROI on both sides of the peak ROI are defined. Some implementations allow one continuum ROI either below or above the peak. It is critical that ROIs of adjacent gamma

ray peaks do not overlap. If this condition is violated, proper subtraction of the continuum underneath the peak ROI will not be possible. This will affect image reconstruction and will bias TGS results.

10.2.3 During system installation, care must be taken to separate the stepper motor electrical cables from the detector cables. This is to avoid any electromagnetic noise interference with the detector signals.

10.2.4 If a rate loss source is used, its position with respect to the detector must be maintained at all times. If the position varies, the accuracy of rate loss corrections would be compromised. A decay correction is needed. Periodic measurements may reset the clock so that the decay factor does not introduce uncertainties.

11. Procedure

11.1 Ensure that the TGS system is configured to assay using the correct geometry and use the calibration for the given assay geometry.

11.2 Set up the software to assay the container over the desired number of vertical layers. Set the assay time for the transmission and emission scans.

11.3 Load the item on to the rotating and translating platform. The container must be loaded such that it is within the scan diameter set up for the assay.

11.4 Start the assay. At the beginning of the assay, the rotator assembly with the container loaded on it moves to a position clear of the detector to transmission source line of sight. The transmission source is exposed to the detector and gamma-ray counts, unattenuated by the item, are measured. This measurement not only acts as an energy and efficiency calibration check, but also helps to determine the transmission beam intensity directly, that is without the need to apply a calculated decay correction.

11.5 The item is scanned in 3 degrees of freedom; rotational, translational, and vertical. At each vertical layer a transmission scan and an emission scan are performed. The container is continuously translated and rotated during the scans performed at each layer.

12. Data Analysis

12.1 Tomographic transmission and emission gamma scans are acquired of characteristic gamma rays. See Table 1 for energies of primary isotopes to be measured with associated transmission and rate-loss correction sources. The transmission and emission scans are performed as described in Section 11.5. The tomographic analysis is performed using the dedicated software package. This analysis can be broken down into five stages: *(1)* transmission image reconstructions, *(2)* construction of an attenuation-corrected emission response matrix, *(3)* emission image reconstruction, *(4)* normalization of emission images to the measured total count rates (optional), and *(5)* summation of emission image voxel values. For each peak assayed, the sum of the emission image voxel values gives the uncalibrated source strength for that radionuclide.

12.1.1 The description of the transmission problem requires a logarithmic conversion to obtain a linear form. Let pi equal the i^{th} transmission measurement:

$$p_i = counts_i / counts_{max} \quad (5)$$

where $counts_i$ is the photon count at a given gamma ray peak energy in the i^{th} transmission measurement and $counts_{max}$ is the unattenuated count at the same gamma ray energy of the transmission source. We define the logarithmic transmission, v_i, by the relation:

$$v_i - ln(p_i) \quad (6)$$

With this conversion, the transmission problem can be described by an n_{views} by n_{voxels} thickness matrix T, where each element T_{ij} is the linear thickness of the j^{th} voxel along a ray connecting the transmission source and the detector in the i^{th} measurement position. The transmission image is found as the solution of the linear system:

$$\bar{v} = \bar{T} \cdot \bar{\mu} \quad (7)$$

where v is a n_{views}-vector of logarithmic transmission measurements and μ is a n_{voxels}-vector of linear attenuation coefficients.

12.1.2 The analysis software performs transmission image reconstruction to create an image of the attenuation coefficients for each voxel in the item at every transmission gamma ray peak energy. Each layer of the item is solved independently. The transmission peak energies are usually different from the emission peak energies. The attenuation coefficient information at the transmission energies must be converted to the emission energies. A "material basis set" method is used to determine the average linear attenuation coefficient in each voxel. In this method, the attenuation coefficients for any matrix material are solved in terms of 2 or 3 elements spanning the expected Z range (for example, boron and lead). The energy independent partial densities for the low Z and high Z components are determined in each voxel. A library of mass attenuation coefficients is required. Also required are transmission data at two or more gamma ray energies, preferably spanning the low (for example, 122 keV) and high energy (for example, 1408 keV) regimes. If such an energy spread cannot be achieved using a given external transmission source, then prior knowledge of the matrix composition or a representative atomic number (Z) value is required to solve for the linear attenuation map. The NNLS algorithm is commonly used to obtain an initial estimate of the transmission map.

12.1.3 The data form needed for emission imaging is the net count rates of the gamma ray peaks emitted by the radionuclides to be assayed. Deadtime corrections must be applied on a per grab basis in TGS analysis, for every ROI. This correction is typically done using either a rate loss gamma ray source or a rate loss pulser. In either case, a reference peak of known true rate is added to every spectrum in every grab. In each data grab, the known true rate of the rate loss peak divided by the measured rate gives the dead time correction for all other peaks in that data grab. For each view, this rate is a sum of individual rate contributions from potentially every voxel in the item. The emission analysis can be mathematically described as follows—the attenuation-corrected emission image is found as the solution of the linear system:

$$\bar{d} = \bar{F} \cdot \bar{s} \quad (8)$$

where d is a n_{views}-vector of measurements and s is a n_{voxels}-vector describing the emission source intensity distribution. The F matrix is defined as the attenuation-corrected efficiency matrix. The elements of F are given by the relation:

$$F_{ij} = E_{ij} \cdot A_{ij} \quad (9)$$

where A_{ij} is the fractional attenuation, due to the drum contents, of photons emitted from the j^{th} voxel in the i^{th} emission measurement. The E matrix is the geometric efficiency matrix for the given assay geometry. The elements of the geometric efficiency matrix are calculated by defining the gamma ray paths to the detector from each voxel in each view of the emission scan. The geometric efficiency depends on the solid angle subtended by each voxel in each view and the distance to the detector, and is independent of energy. The values of A_{ij} are estimated from the transmission image using Beer's Law:

$$A_{ij} = \Pi_k exp(-t_{ijk}\,\mu_k) \quad (10)$$

where the triply-indexed quantity t_{ijk} is the linear thickness of the k^{th} absorbing voxel along a ray connecting the j^{th} emitting voxel and the detector in the i^{th} measurement position. If the k^{th} voxel is not on a line between the emitting voxel and the detector, t_{ijk} is zero. While the table of t_{ijk} values is constant, A depends on the drum contents and must be computed anew for each drum assayed. Thus, for each emission energy of interest an attenuation-corrected emission response matrix is constructed by multiplying the unattenuated response matrix elements by the fractional attenuation losses for each view and voxel. In the absence of an attenuating item matrix, the emission response matrix elements represent relative counting efficiencies (that is, detection probabilities) for each voxel position in each view. The effects of collimation and distance to the detector are included, but not intrinsic detection efficiencies. Therefore, the energy dependence of the efficiency is not accounted for at this stage and the uncorrected emission response matrix is the same for all emission energies. The emission image for each gamma ray energy is reconstructed from the emission data and the attenuation-corrected emission response matrix for that energy. Several image reconstruction algorithms have been used in TGS analysis and have been found suitable. These include the Algebraic Reconstruction Technique (ART) (**5**), the Expectation Maximization (EM) Algorithm (**6**, **7**), and other methods. Unlike the transmission images, in which adjacent layers can be considered to be independent, adjacent layers in the emission problem are highly coupled. Because of this strong layer coupling, emission imaging is done 3-dimensionally on the entire item rather than one layer at a time as in transmission imaging.

12.1.4 The emission images determined in the previous step are normalized so that the sum of the reverse projected count rates, based on the reconstructed image, equal the actual total measured rate at each energy. The reason for this normalization is to circumvent the so-called "low mass bias" problem (see 13.2.5 for more details). Most TGS systems in operation today use this approach. In the equation below, the radionuclide mass or activity, M, is the product of K, the calibration constant, N, the normalization factor discussed above and the summation of attenuation-corrected emission matrix elements over all voxels.

$$M = K \cdot N \cdot \sum_i s_i \quad (11)$$

12.1.4.1 The individual emission image voxel values are summed to give the total uncalibrated source strength for each emission gamma ray analyzed. Note that if the normalization in the previous step is performed, this sum will have already been computed. This is the end result for the tomographic portion of the analysis. Subsequent handling of the results is similar to that used in other gamma ray assay methods.

12.2 In the event that a single radionuclide 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 radionuclide/total ratio to the radionuclide assay value to determine the total element content. TGS analysis does not determine isotopic ratios. Vendor-supplied software options may allow separate isotopic composition analysis. See Test Method C1030 for a discussion on isotopics.

12.3 A bibliography of the TGS technique is given in (**10**).

12.4 The following diagnostics may be useful in an expert review of suspicious analysis results.

12.4.1 Examine the reports generated by the analysis software and address any warning or error messages (or both). For example, if the energy and shape calibration had shifted, the ROI counts may be incorrectly assigned to a different gamma ray energy. Also, the analysis might miss the reference peak ROI and an incorrect rate loss correction will be applied. Analysis reports generated by commercially available TGS software packages typically flag these situations and warn the user.

12.4.2 For those radionuclides that emit more than one gamma ray energy, compare the TGS results (mass or activity) for the different gamma lines from a given radionuclide (for example, 129 keV and 414 keV gammas from ^{239}Pu). If the attenuation correction has been calculated and applied correctly, the TGS results for different gamma ray lines from a given radionuclide must agree within the uncertainty bounds. If the results from different gamma ray lines are inconsistent, it could be due to self-attenuation. A lump correction may be required. The TMU may need to be expanded.

12.4.3 Visually examine the transmission and emission images generated by the TGS analysis software. In the transmission image, look for the presence of artifacts such as the "checker board" pattern where adjacent voxels are imaged as completely black or completely white in an alternating fashion. This is usually because of poor statistical precision in the transmission grab data. In the case of emission images, look for loss of contrast or sharpness in regions where source concentrations are indicated. This could once again be due to poor statistics in the emission grab data. A longer assay time may be needed.

12.4.4 Where possible it is good practice to compare the transmission image of the container matrix to what is known from process knowledge or real time radiography (RTR) data, if these are available.

12.4.5 Retrieve the grab data for all layer scans and for several (or all) transmission and emission energies. For a given layer scan, and a given gamma ray energy, plot the counts registered in each grab as function of the grab number. These plots are referred to as "sinograms" because of their sinusoidal shape. Examining these plots will provide valuable diagnostics regarding mechanical movement of the platform and also pulse processing electronics. Abnormally high or low counts in the

grab data or a deviation from the expected sinusoidal behavior are indicative of problems.

13. Measurement Uncertainty

13.1 *Precision*—Precision of a TGS system is defined as the relative standard deviation of the results of multiple assays performed using a given representative item. The factors affecting the precision of TGS assay are counting statistics, mechanical reproducibility of geometry and scan positions, stability of pulse processing electronics, and the variability of environmental background. Precision of TGS assays of waste containing SNM or non-SNM have been reported in the literature (**10-12**). TGS assays of pyrochemical salt waste contained in short-convenience cans have yielded a precision of ±6.0 % (**10**). In the work described in reference (**10**), the total plutonium masses that were assayed were in the 60 g to 200 g range. TGS assay of low mass levels of ^{239}Pu (0.168 g) have been shown to yield a precision of ±10 % (**11**). A precision of ±2 % has been obtained in TGS assays of low mass levels of ^{238}Pu (0.1 g) as well as in TGS assays of ^{235}U samples with a mass of 31 g (**11**). For non-SNM nuclides such as ^{137}Cs and ^{60}Co, the precision was measured with point sources distributed inside 208 litre drum matrices, with matrix density ranging from 0.4 g.cm^{-3} to 0.7 g.cm^{-3} (**12**). The precision was measured to be in the range between 3.5 % to 7 %. Thus it can be stated that for a TGS system using the current state-of-the-art electrical, mechanical and electronic components, it is possible to achieve a precision of ±2 % to ±10 % at 1σ level of confidence depending on the mass or activity levels being measured and the matrix density.

13.2 Bias is the difference between the measured mass or activity of a radionuclide and the true value. Therefore, the observed bias is a measure of the accuracy of the TGS system. Bias is caused by systematic uncertainties present in the measurement or in the analysis (or both) methods. Bias in TGS assay results (or accuracy of TGS) has been estimated for waste containing SNM and non-SNM nuclides (**1, 11, 13**). TGS assay of a single point-like source of ^{239}Pu (98.9 g) located inside a heterogeneous 208 litre mock waste drum filled with electronic scrap has been shown to be within ±8.4 % at 1σ level of confidence (**1**). The accuracy estimate of ±8.4 % was determined based on 60 assays, with the point like source located at different positions within the drum matrix. In the work described in reference (**1**) good counting statistics were ensured by choosing a relatively high sample mass of 98.9 g. Systematic uncertainties in TGS assay results for pyrochemical salt waste contained in cans has been shown to be ±6.9 % at 1σ confidence level (**11**). This estimate is based on 240 measurements of 108 samples. In the work described in reference (**11**), the TGS results have been compared with corresponding results from calorimetry. For the sample set of 108, the relative deviation of the TGS results with respect to calorimetry was fitted with a Gaussian distribution with a standard deviation of 9.1 %, and yielded a reduced chi-squared value of 0.6. The 9.1 % value was the total measurement uncertainty which included systematic as well as random uncertainties. A reduced chi-squared value of 0.6 confirmed that the observed deviations were consistent with the uncertainty estimates. For non-SNM nuclides such as ^{133}Ba (300 keV – 400 keV), ^{137}Cs, and ^{60}Co, the systematic uncertainties in TGS results have been quantified at least 2 different matrix densities, 0.17 g.cm^{-3}, and 0.61 g.cm^{-3} (**14**). In the work described in reference (**14**), point sources of ^{133}Ba, ^{137}Cs, and ^{60}Co were located inside a 208 litre drum matrix and measured. For the lower matrix density of 0.17 g.cm^{-3}, the systematic uncertainties were ±5.5 % for all energies in the 300 keV to 1332 keV range (**14**). For the moderate density of 0.61 g.cm^{-3} , the systematic uncertainties were ±10.5 % in the 300 keV – 400 keV energy range, ±8.6 % at 662 keV, and ±8.0 % at 1173 keV and 1332 keV (**13**). In the following sub-sections some common sources of uncertainties that could impact TGS assays are discussed.

13.2.1 In some systems, low count rates can introduce a bias which tends to overestimate the radionuclide mass (due to round off errors of approximately negative numbers in the background continuum corrected grab data). This bias can have the effect of increasing the lower end of the acceptable mass range for the system. This effect can be reduced by using longer count times.

13.2.2 *Lumps*—When the radiation emitting material is concentrated in dense lumps within the matrix, the assay results can be biased low because of self-attenuation in the lumps (**13, 15**). Self-attenuation errors are difficult to calculate with confidence, unless the radiation emitting material has measurable gamma rays at multiple energies. Even then, it can be difficult to estimate a valid correction factor due to the variety of shapes and sizes of lump which may be present.

13.2.2.1 For a 1-g lump of plutonium, the worst-case self-absorption error could be as large as 60 %. At this level, the multiple gamma line assays would flag the waste drum as a potential assay problem.

13.2.2.2 For U, the effects occur at lower ^{235}U loadings because the 186-keV gamma ray is so weakly penetrating. For ^{235}U, it is more difficult to identify this as a problem, much less to estimate a correction (**16, 17**). For ^{238}U assay, this is much less of a problem because the 1001-keV gamma ray from the ^{234m}Pa progeny penetrates well.

13.2.3 *Electronic Noise*—The presence of electronic noise can lead to deleterious count rate dependent biases which can render assay results inaccurate. Electronic noise must be minimized as much as possible in order to obtain reliable assay results. Detector cables must be isolated or shielded from all potential noise sources.

13.2.4 *Partial Volume Effects*—In a waste matrix where there is a significant non-uniformity within a given vertical layer, it is possible that the highly-collimated transmission beam does not sample a representative volume of the voxel. This will result in a bias in the attenuation values that are calculated for the voxels in the given layer. For example, if the drum is not filled with a matrix material up to the brim, the topmost layer will only be partially full. The transmission beam may only traverse the filled portion of the layer, thus resulting in higher attenuation values for the voxels in the top layer. The bias could be in the 10 %–30 % range depending on the gamma ray energy.

13.2.5 *Low Mass Bias*—The phrase "low mass bias" has been informally used in TGS assays to describe systematic errors that arise from zero truncation in the emission image reconstruction. Emission image reconstruction algorithms used in TGS are constrained to give a non-negative result in every image voxel. Therefore, repeated assays of items with low masses or activities can give results that are high on average. The effect is strictly dependent on the signal-to-noise ratio of the item and can, therefore, also be exacerbated by a high continuum background below the peak of interest. One TGS methodology circumvents this problem by requiring that the image reconstruction algorithm preserve the total count rate in reverse projection (**18**). This is achieved by a simple normalization of the mass image. If an image reconstruction algorithm used in some TGS method does not address and correct for the low mass bias problem, then an appropriate error due to this effect should be added to the TMU.

13.2.6 Extrapolation of transmission measurements to energies significantly beyond measured values, too low or too high, can lead to biased results.

13.3 *Calibration Error*—The accuracy of TGS mass or activity calibration is limited by the uncertainties quoted by the manufacturer of the calibration standards used. Any bias in the calibration standard will directly impact the assay results. Poor statistical precision of the calibration data (for example, 5 %) will also have a significant impact on the accuracy of calibration. Gamma ray interferences on calibration data will skew the calibration results. It is prudent to subtract environmental background from emission and transmission data.

13.4 *Geometry Error:*

13.4.1 *Alignment Errors*—Misalignment in TGS can be defined as the difference between the actual geometry of the scanner and the assumed geometry used to compute the transmission and emission response matrices for image reconstructions. Misalignments can lead to a significant assay bias.

13.4.2 *Source*—If the axis of the item is offset with respect to the center of rotation (for example, a wobbly drum), the resulting emission image will be displaced with respect to its correct location. This will cause a bias in the assay results.

13.4.3 *Mechanical*—A transverse offset between the detector/collimator center line and the item center of rotation in the presumed x=0 position can lead to significant biases in the assay results. For example, an offset of 1 cm can introduce a 6 %–8 % bias.

13.4.4 *Collimator*—A pitch of the transverse motion direction relative to the x-axis (which is defined to be normal to the collimator axis) in the horizontal XY plane can lead to an offset which can bias the assay results.

13.4.5 *Detector*—An incorrect specification of the virtual detector offset that gives the distance between the detector end cap and crystal plus the mean penetration depth of the photons can bias the efficiencies high or low and lead to inaccurate assay results.

13.5 *Counting Statistics in Transmission and Emission Grabs*—The statistical uncertainties in the TGS numbers may be calculated using the Monte Carlo Randomization (MCR) Method (**19**). The MCR method is adopted since no general closed form error propagation formulae are known for EM, ART, and NNLS reconstruction algorithms. In this method, data is randomized using Poisson statistics to generate N replicate data sets that have a high probability of being representative of N actual replicate measurements. The measured counts in each spectral ROI are randomized in each view (or data grab) for both the transmission and emission scans. The randomization is performed on each peak ROI as well as on the background ROIs to the right and left of each peak. Several such replicate analyses are performed using randomized data and the standard deviation in the TGS is calculated. The standard deviation is deemed to be representative of the statistical uncertainty in the measured TGS results. It is recommended that at least 20 replicate trials be used to estimate the statistical uncertainties. One of the drawbacks of the MCR method is an increase in the computation time required to complete the TGS analysis. However, using a 2 GHz Pentium processor, a TGS analysis including the MCR error estimates for a set of 3–4 energies can be completed in less than 5 minutes. This is significantly shorter than the time taken to perform actual repeat measurements and one can anticipate faster machines in the future. It is recommended that the statistical uncertainty is estimated for all assays as part of the overall uncertainty estimate.

13.6 *Error Propagation*—The biases must be eliminated where possible to maintain a small assay uncertainty. For example, the bias due to mechanical alignment or geometry inconsistencies can be minimized or eliminated by careful installation of the system. Another example of a bias that can be eliminated is the "low mass bias." However, other bias components such as those due to lumps and partial fill heights cannot be easily corrected and therefore must be estimated as part of a TMU analysis.

13.6.1 Not all error terms may be described using a Gaussian function and some terms can be asymmetric. For example, the error due to lumps, which is essentially a self-attenuation effect, tends to under report the mass or activity (a one-sided bias). Therefore, one must exercise caution in combining error terms and treat positive and negative terms separately. The positive error terms may be added in quadrature and the square root taken. Negative error terms can be treated similarly. This allows the final uncertainty to be asymmetric about the mean value (**20**).

13.6.2 The overall measurement uncertainty can be estimated by assaying source distributions and surrogate drum matrices that are representative of real items. One approach would be to randomly distribute "x" number of point sources (x=3 typically for a 208L drum) of a given radionuclide inside a surrogate matrix and perform an assay. Assays are performed for 20 to 30 such random distributions of the x point sources in each of the representative matrices. For each matrix, the minimum and maximum response values are determined. The point source distribution error is assumed to follow a Gaussian distribution and the difference between the minimum and maximum responses is taken to be the uncertainty at $\pm n\sigma$ limits. This exercise must be repeated for a series of surrogate matrices that span the density range of interest and represent

the heterogeneous nature of real items. The measurement uncertainty must also be determined as a function of gamma ray energy.

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