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Standard Guide for Nondestructive Assay of Special Nuclear Material Holdup Using Gamma-Ray Spectroscopic Methods[1]

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1. Scope

1.1 This guide addresses methods used to prepare for and to perform, using gamma-ray measurements, the nondestructive assay (NDA) of radioisotopes, for example, ^{235}U, or ^{239}Pu, remaining as holdup in nuclear facilities. Holdup occurs in facilities where nuclear material is processed. This guide includes the measurement of holdup of Special Nuclear Material (SNM) in places where holdup may occur, such as in process equipment, and in exhaust ventilation systems. This guide includes information useful for management planning, selection of equipment, consideration of interferences, measurement program definition, and the utilization of resources.

1.2 The measurement of nuclear material help up in process equipment is both an art and a science. It is subject to the constraints of politics, economics plus health and safety requirements, as well as to the laws of physics. The measurement process often is long and tedious and is performed under difficult circumstances of location and environment. The work combines the features of a detective investigation and a treasure hunt. Nuclear material held up in pipes, ductwork, gloveboxes, heavy equipment, and so forth, usually is distributed in a diffuse and irregular manner. It is difficult to define the measurement geometry, identify the form of the material, and measure it without interference from adjacent sources of radiation. A scientific knowledge of radiation sources and detectors, calibration procedures, geometry and error analysis also is needed **(1)**.[2]

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

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

C 1009 Guide for Establishing a Quality Assurance Program for Analytical Chemistry Laboratories Within the Nuclear Industry[3]

C 1030 Test Method for Determination of Plutonium Isotopic Composition by Gamma-Ray Spectroscopy[3]

2.2 *ANSI Standards:*

ANSI N15.37 Guide to the Automation of Nondestructive Assay Systems for Nuclear Materials Control[4]

ANSI/ASME NQA-1-1983 American Nuclear Society Requirements for Nuclear Power Plants[4]

2.3 *U.S. Nuclear Regulatory Commission Regulatory Guides:*

Regulatory Guide 5.23, In Situ Assay of Plutonium Residual Holdup[5]

Regulatory Guide 5.9, Rev 2, Guidelines for Germanium Spectroscopy Systems for Measurement of Special Nuclear Material[5]

3. Terminology

3.1 *Definitions:*

3.1.1 *absorber foils, n*—thin foils, usually of copper, tin, cadmium, or lead, used to intentionally attenuate the gamma flux reaching a detector. Absorber foils, typically, are used to reduce the counting rate from low-energy gamma rays not needed for the measurement.

3.1.2 *attenuation, n*—reduction of measurable gamma-ray flux due to the interaction of gamma rays with the container, holdup and other material between the source of the gamma-rays and the detector.

3.1.3 *attenuation correction, n*—a correction to the measured count rate that enables one to make an estimate of the actual gamma-ray emission rate from the holdup, thereby correcting for the attenuation effects of the measurement situation.

3.1.4 *background, n*—any count in a gamma-ray peak, which did not originate as a gamma ray at the assay energy in the sample or item being measured, can be considered background. The three main contributors to background are as follows:

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

[3] *Annual Book of ASTM Standards*, Vol 12.01.

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

[5] Available from the U.S. Nuclear Regulatory Commission, Washington, DC, 20555.

3.1.4.1 *Compton scattering, v*—which produces a continuum under the peak of interest due to scattering of higher energy gamma rays;

3.1.4.2 *Peaked background*—Gamma rays of the assay energy, which originate in sources other than the holdup being measured; and,

3.1.4.3 *Summed background, n*—Nonpeaked counts under the peak of interest that result from the summing of lower energy gamma rays, or Compton events, or both.

3.1.5 *collimated detector*, *n*—a detector surrounded by a shield that imposes a directional response on the collimated detector. The shield, called a collimator, generally is a cylinder of high-Z material, for example lead, tungsten) mounted coaxially to the detector and extending over the detector and beyond the detector face. Since a collimator is designed to be used with and affects the calibration of a specific detector, it is appropriate to refer to the unit as a detector-collimator assembly.

3.1.6 *contact measurement*, *n*—a special case of a near-field measurement in which measurements are made with the detector assembly in contact with the item, for example, tank, pipe, ductwork, being assayed.

3.1.7 *far-field measurement*, *n*—measurement with a detector and collimator such that the assumptions for a generalized geometry assay are valid **(1)**.

3.1.8 *field of view*, *n*—the entire range encompassed by the collimated detector when it is trained in a particular direction.

3.1.9 *holdup*, *n*—residual special nuclear material in processing or support equipment areas.

3.1.10 *infinite thickness*, *n*—the thickness of material through which the gamma rays of the designated energy cannot penetrate **(2)**; however, for the purposes of this guide, the thickness through which 99.9 % of the gamma rays of the designated energy cannot penetrate, will be used.

3.1.11 *isotopic mapping*, *v*—use of high resolution gamma-ray spectrometry to identify gamma-ray emitting isotopes and interfering gamma rays at representative locations on the measurement items.

3.1.12 *near-field measurement*, *n*—measurement made at a detector to holdup distance such that the far-field assumptions are not satisfied.

3.1.13 *scan method*, *n*—rapid, that is, short-count time, measurement at specific locations or movement of a gamma-ray count rate meter along process equipment to qualitatively identify the presence of radioactive material above a predetermined activity level (“hot spots”). It can be used to map the extent of areas with a similar activity level or to identify an area of maximum activity.

3.1.14 *self attenuation*, *n*—attenuation of gamma rays produced within the holdup by the holdup itself.

3.1.15 *shadow shield*, *n*—attenuating material placed between the shielded detector and radiation sources not part of the assay item so as to limit the contribution from those extraneous sources to the observed measurement or background count rates.

3.1.16 *shielded detector*, *n*—a detector surrounded on all surfaces but one with material that provides significant attenuation of gamma-rays.

3.1.17 *transmission correction*, *n*—an attenuation correction is determined using a gamma-ray emitting source, sometimes a transmission source is used, placed behind the holdup with respect to the detector.

3.1.18 *working source*, *n*—an item containing, in a fixed geometry, a fixed quantity of a radioisotope to be measured. A working source can be used for routine measurement control checks if the gamma-ray emission rate is well characterized.

4. Summary of Guide

4.1 *Introduction*—Holdup measurements range from the assay of a single item to routine measurement of a piece of equipment, to an extensive campaign of determining the total SNM in-process inventory for a processing plant. Holdup measurements differ from other nondestructive measurement methods in that the assays are performed in situ on equipment associated with the process. Often, the chemical form and geometric distribution of the SNM are not known. These unique challenges require for each measurement a specific definition of what is expected from the assay, specific information about the item or items to be assayed, design of the assay, and special preparation for the assay. The amount of effort expended and level of detail attained for each of these preparatory activities is dependent on both assay requirements and available resources.

4.2 *Definition of Requirements*—Definition of the holdup measurement requirements should include, as a minimum, measurement goals, for example, criticality control, SNM accountability, security, time constraints for the measurements, resources available, for example, personnel, equipment, funding, and desired measurement sensitivity, accuracy, and uncertainty.

4.3 *Information Gathering and Initial Evaluation*—Information must be gathered concerning the item or items to be assayed and the level of effort needed to meet the holdup measurement requirements. Preliminary measurements may be needed to define the location and extent of the holdup, to determine the SNM isotopic composition or enrichment, and to identify potential interfering isotopes. Factors to be considered include the geometric configuration of the item or process equipment to be assayed, location of the equipment in the facility, attenuating materials, sources of background or interferences, safety considerations (both radiological and industrial) associated with the assay, plus the personnel and equipment needed to complete the assay. Sources of information may include a visual survey of the items, engineering drawings of the item and other equipment in the vicinity, process knowledge, and prior assay documentation.

4.4 *Task Design and Preparation*—The initial evaluation serves as the basis for choosing the quantitative method, assay model, and subsequently, leads to determination of the detection system and calibration method to be used. Appropriate standards and support equipment are developed or assembled for the specific measurement technique. A measurement plan should be developed. The plan may outline required documentation, operating procedures, including background measurement methods and frequencies, plus training, quality and measurement control requirements. Necessary procedures, including those for measurement control, should be developed,

documented, and approved.

4.5 *Measurements*—Perform measurements and measurement control as detailed in the measurement plan or procedure.

4.6 *Evaluation of Measurement Data*—Appropriate to the quantitative method chosen, corrections are made for gamma-ray attenuation effects, for example, the container, item matrix, absorbers, and measured background. These corrections are applied in the calculation of the assay value. Measurement uncertainties are established based on factors affecting the assay.

4.6.1 Converting measurement data to estimates of the quantity of nuclear material holdup requires careful evaluation of the measurement against calibration assumptions. Depending on the calibration and measurement methods used, corrections may be necessary for geometric effects (differences between holdup measurement and calibration geometries), gamma-ray attenuation effects, background, and interferences. Measurement uncertainties are estimated based on uncertainties in assay parameters, for example, holdup distribution, attenuation effects, measured count rates.

4.6.2 Results should be evaluated against previous results or clean out data, if either is available. If a discrepancy is evident, an evaluation should be made. Additional measurements with subsequent evaluation may be required. The assay should be documented.

4.7 *Documentation*—Measurement documentation should include a description of measurement parameters considered important to the calibration and measurement techniques used, estimated precision and bias, and comparison to other measurement techniques.

5. Significance and Use

5.1 The following methods assist in demonstrating regulatory compliance in such areas as safeguards SNM inventory control, criticality control, and decontamination and decommissioning (D&D). This guide can apply to the measurement of holdup in equipment whose gamma-ray absorption properties may be measured or estimated. These methods may be adequate to accurately measure items with complex distributions of holdup and attenuating material, however, the results are subject to larger measurement uncertainties than measurements of less complex distributions of holdup.

5.2 *Scan*—The scan method is used to provide a description of the extent, location, and the relative quantity of holdup. It can be used to plan quantitative assays. In addition, the method can be used in combination with quantitative measures to estimate holdup quantities in large pieces of equipment, for example, long pipes or ductwork.

5.3 *Isotopic Mapping*—Isotopic mapping is used to estimate the relative isotopic composition of SNM at specific locations. It can be used to detect the presence of isotopes that emit radiation, which could interfere with the assay. The measured isotopic composition may represent the average composition of the material closest to the detector. If the holdup is not isotopically homogeneous, the measured isotopic composition will not be a reliable estimate of the bulk isotopic composition.

5.3.1 *Enrichment Measurements*—A special case of the determination of isotopic abundance is the measurement of the ratio of two isotopes. Generally, this is applied to uranium.

5.4 *Quantitative Measurements*—These measurements result in quantification of the mass of SNM in the holdup. They typically include all the corrections, such as attenuation, and descriptive information, such as isotopic composition, that are available concerning the holdup.

5.5 *Spot Check and Verification Measurements*—Periodic measurement of holdup at a defined point can be used to detect or track relative changes in the holdup quantity over time. Either the scan method or a quantitative method can be used.

5.6 *Indirect Measurements*—Quantification of an isotope by measurement of a daughter isotope or of a second isotope if the ratio of the abundances of the two isotopes is known. This can be used when there are interfering gamma rays or when the parent isotope does not have a sufficiently strong gamma-ray signal to be readily measured. If this method is employed, it is important that the ratio of the two isotopes be known with sufficient accuracy to meet the holdup measurement quantification requirements.

5.7 *Mathematical Modeling*—An aid in the evaluation of complex assay situations. Actual measurement data are used with a mathematical model describing the physical location of equipment and materials.

6. Interferences

6.1 *Peaked and Compton Background*—Background can cause problems in several ways.

6.1.1 Peaked backgrounds that fluctuate, for example, a cyclical process or a rotating attenuator, which shields some source of background, during the measurements will cause biased results.

6.1.2 If a background activity (peaked or Compton) is large relative to the gamma-ray flux from the holdup, the overall assay sensitivity will be reduced and uncertainty increased. Small quantities of holdup may be underestimated or missed altogether.

6.1.3 *Sum Peak and Random Summing*—Gamma-ray interactions, such as summing of lower energy gamma rays or summing of the assay gamma ray with another gamma ray, within the detector may produce a change in the observed count rate in either the measurement or background regions of interest. This effect can cause a bias in the measurement results.

6.2 *Peaked Interferences*—Gamma-rays emitted by nuclides other than the nuclide of interest may produce an interference. The nature and magnitude of the interference will depend upon the energies of the gamma ray of assay interest, the interfering gamma ray, and the detection system being utilized. The user will need to assess site specific gamma-ray interferences. Plutonium interferences are discussed in NRC Regulatory Guide 5.23.

7. Apparatus

7.1 General guidelines for selection of detectors and signal-processing electronics are discussed in Guide C 982 and NRC Regulatory Guide 5.9, Rev. 2. Additional guidance for the selection of detectors is given in Test Method C 1030. Data acquisition systems are considered in ANSI N15.37 and NRC Regulatory Guide 5.9, Rev. 2.

7.2 The apparatus chosen for measurements must have

capabilities appropriate to the requirements of the measurement being performed. For example, in order to locate holdup by scanning, a simple system based on a gross gamma-ray detector, for example, a Geiger-Mueller tube, is adequate for some applications. Other applications, where severe interferences or absorption are expected, may require a high-resolution Ge-detector-based system. The quality of assay results are partially dependent upon the capabilities of equipment. The user should choose a suitable trade-off between detector energy resolution, detection efficiency, equipment complexity and equipment size.

7.3 *Scan Measurement Systems*—The minimum gross gamma-ray detection system may be a survey meter. If limited energy discrimination is required a low resolution scintillation detector may be used, such as a bismuth germanate oxide (BGO) or NaI detector, with associated power, signal amplification and scaling electronics. The detection system may be as complex as a Ge-detector/MCA system.

7.4 *Low Resolution Measurement Systems*—Quantitative holdup measurement may be performed using instrumentation that offers portability and simplicity of operation. The instrumentation typically includes a low resolution scintillation detector with spectroscopy electronics in a portable package. Stabilization may be necessary to compensate for electronic drift. At least two energy windows are recommended: one for the peak or multiplet of interest, and another to determine the Compton continuum (background) under the peak. With a low resolution system there may be an adverse impact on measurement bias and precision when compared to a high resolution system.

7.5 *High Resolution Measurement Systems*—A high resolution gamma-ray spectrometry system includes a germanium detector with associated high voltage, signal processing, and data storage electronics. Germanium detectors have sufficient resolution to resolve most types of spectral interferences or allow the use of computer software that will resolve closely spaced gamma-ray peaks.

7.6 *Detector Collimation and Shielding*:

7.6.1 A collimator is used to limit the field of view of a detector so that gamma radiation from the intended source can be measured in the presence of background radiation from other sources. A collimator of appropriate design is important to making accurate holdup measurements.

7.6.1.1 Design of a collimator generally involves arriving at a compromise among several attributes. Among these are a manageable collimator weight versus adequate shielding against gamma rays from off-axis directions, and a fixed acceptance solid angle that is likely not ideal for all measurement situations. Since a collimator is designed to be used and calibrated with a specific detector, it is appropriate to refer to the unit as a detector-collimator assembly.

7.6.1.2 In general, it is not feasible to use more than one detector-collimator assembly during a series of measurements, but special measurement campaigns might require using multiple detector-collimator assemblies with different attributes. Also, any changes in the absorber foils or detector field of view will require recalibration.

7.6.2 Additional shielding may be used to reduce the background incident on the detector from identified nearby sources. For example, attenuators can be placed between the location of interfering gamma-ray activity and the detector.

7.6.3 Absorber foils may be needed to reduce the contribution of low-energy gamma rays to the overall count rate, especially in the assay of ^{239}Pu. For example, foils can be used to reduce high count rates, which can produce spectral distortions and biases in the assay results.

7.7 *Data Processing and Storage*—Use of computers may be desired while conducting holdup measurements. Portable multichannel analyzers provide for some data reduction and storage. Portable computers can be used for increased data reduction and storage capacity.

7.8 *Detector Positioning Apparatus* may be used.

8. Hazards

8.1 *Safety Hazards*:

8.1.1 Holdup measurements sometimes need to be carried out in areas with radiological contamination or high radiation. Proper industrial safety and health-physics practices must be followed.

8.1.2 Gamma-ray detectors may use power-supply voltages as high as 5 kV. The power supply should be off before connecting or disconnecting the high-voltage cable.

8.1.3 Collimators and shielding may use materials, for example, lead and cadmium, which are considered hazardous, or toxic, or both. Proper care in their use and disposal are required.

8.1.4 Holdup measurements often require performing assays in relatively inaccessible locations, as well as, elevated locations. Appropriate industrial safety precautions must be taken to ensure personnel are not injured by falling objects or that personnel do not fall while trying to reach the desired location.

8.2 *Technical Hazards*:

8.2.1 High gamma-ray flux generally will cause pulse pileup, which affects the observed energy and resolution of the peaks, as well as, the total counts observed in the peaks due to summing effects. Extremely high activity holdup may saturate the electronics of certain types of preamplifiers resulting in no counts being registered by the equipment.

8.2.2 Electronic instability in the signal processing electronics, can cause shifts in the spectrum, which will significantly alter the assay results. Electronic instability is most pronounced for NaI systems. Unconditioned, unfiltered power supplies affect electronic stability.

8.2.3 *Secular Equilibrium*—If the gamma ray from a daughter isotope is used to quantify holdup, such as with ^{238}U and^{234m}Pa, secular equilibrium within the holdup should be verified. The results will be understated if secular equilibrium is not reached. The results will be overstated if secular equilibrium is not reached in holdup remaining following chemical treatment that preferentially removes the parent isotope.

8.2.4 *Infinitely Thick SNM Holdup*—If the holdup is infinitely thick to the measurement gamma rays, transmission corrections are not possible and the measurement results will be biased low. An alternative method, such as using higher energy gamma rays or using neutron measurement techniques

or holdup sampling coupled with destructive assay, might be considered.

9. Measurement Plan Development

9.1 *Measurement Program Requirements*—Prior to the evaluation of an assay situation, specific information must be gathered regarding what is expected of the measurement or measurement program. The information should provide the boundaries for the task or project. This information typically includes the following:

9.1.1 Identification of item or piece of equipment to be measured.

9.1.2 Isotope or isotopes of interest.

9.1.3 Acceptable level of measurement uncertainty.

9.1.4 Acceptable lower detection limit for the assay.

9.1.5 Intended and potential applications for results, for example, criticality risk assessment, SNM accountability, etc.

9.1.6 Administrative requirements, for example, quality assurance requirements, documentation and reporting requirements.

9.2 *Constraints*:

9.2.1 The time available to perform the measurement(s), that is how long before a report or compilation of data is required.

9.2.2 Resources available to perform the individual measurement or the measurement program.

9.3 *Personnel and Procedures*:

9.3.1 Since holdup measurements are made with less sample preparation and under a wider range of conditions than other measurements, it is more important that formal procedures be developed for the assays. Procedures can incorporate lessons learned from previous experience including both problems plus their resolution and insights or technique improvements.

9.3.2 Personnel performing holdup measurements must have adequate training, education, and experience. Initial measurements generally require much more expertise than routine measurements, which can be performed by trained personnel using established procedures.

9.4 *Safety Considerations*—Evaluation and mitigation, if possible, of radiological and industrial safety issues must be performed prior to initiating measurements.

9.5 *Facility Evaluation*—The objective of the evaluation is to develop a measurement plan. This consists of several activities, which are difficult to perform sequentially. Some are performed in parallel and iteration often is helpful. Each assay situation is unique. Information must be gathered and evaluated concerning the item or items to be assayed, as well as, concerning the level of effort necessary to obtain the required level of quality and precision for the assays.

9.5.1 Inspect the area(s) and/or equipment to be assayed to gain an overview of the task at hand. Consider measurement geometry, other sources of radiation, attenuating materials, and the physical location of the item or equipment.

9.5.2 If possible, interview any personnel who may be familiar with the area(s) or equipment to be assayed during the measurement campaign. They may be able to provide first-hand information on current and historical process information, and other potentially invaluable insights for considerations noted in Sections 8 and 9.5.1. Also, process operators and management that have participated in previous clean out campaigns and maintenance projects may be a valuable resource in determining the location and characteristics of holdup.

9.5.3 Obtain accurate engineering drawings, if available, to determine areas of probable SNM holdup. The drawings are useful during the identification of measurement locations, determination of physical measurement techniques and development of attenuation corrections.

9.5.4 Obtain information, such as the process flow sheet, regarding the process or processes employed in the area(s) to be assayed. Determine the status of the facility, whether it is in operation or shut down. Assure that there will be no movement of SNM during measurements of process components.

9.5.5 Determine what isotopes are present. Determine whether the relative isotopic distribution remains constant throughout the areas to be assayed. This will include the isotopes of interest as well as interfering radionuclides. Assess whether the issue of secular equilibrium will be a factor.

9.5.6 Scan measurements can be performed to locate areas that will later be measured quantitatively. The scan information also can be used to assess the size and complexity of the task. Locations of holdup exceeding a predetermined activity level are noted for later quantitative measurements.

10. Develop Measurement Strategy

10.1 A critical step in the evaluation process is the determination of how the measurements will be performed. For each measurement location, this involves deciding what calibration models will be used to relate detector response to SNM content, how to minimize or measure background and interfering radiation, plus how to determine attenuation corrections for each measurement location.

10.2 Several measurement techniques may be used. Each technique has advantages and disadvantages, which must be evaluated in light of specific assay situations and availability of physical standards and measurement equipment. Resolution of these issues can be an iterative process to arrive at a strategy which optimizes the ability to determine the holdup quantities given the constraints on the effort **(3,4)**.

10.3 Selection of assay calibration models includes assessment of factors like the geometric configuration of the process equipment to be assayed, an estimate of how the SNM is distributed, the location of the equipment in the facility, safety considerations (both nuclear and nonnuclear), and information available from historical data.

10.3.1 A calibration model using a far-field geometry **(5)** generally provides accurate results. Far field measurements are less sensitive to how the SNM is distributed than, for example, a near-field geometry. Interferences or attenuation problems may require use of contact or near field measurement models. A simple, item specific model may allow results to be reached rapidly with minimal analysis and with acceptable accuracy.

10.4 *Selection of Measurement Techniques*—Other factors that are generally required for gamma-ray measurements are selection of an assay gamma-ray or band of energies, attenuation correction for both holdup thickness and container

thickness, distance between the source and the detector, distance between contiguous measurements, and angle between the source and the detector.

10.4.1 *Selection of Assay Gamma-Ray Energy*—Higher energy gamma rays transmit through intervening materials better, but lower energy gamma rays often are more prolific for SNM isotopes of interest. Consideration must be given to what other gamma emitters are present in the facility as either background or interferences. Energy compromises may be necessary to obtain adequate count rates, or avoid interferences, or both. Table 1 lists useful gamma-ray energies for several isotopes along with their specific activity. Table 2 gives common interferences for the Table 1 gamma-ray energies.

10.4.2 *Attenuation Correction*—Estimates of attenuation correction factors for both the container wall and the material matrix (self-attenuation) must be determined. Available methods for estimating container attenuation corrections are as follows:

10.4.2.1 Compute correction factors using the wall thickness, determined from plant drawings or ultrasonic thickness measurements, and published linear or mass attenuation coefficients **(6)**.

10.4.2.2 Measure the transmission using an external radiation source **(7)**.

10.4.2.3 Multiple gamma-ray energies from the nuclide in the sample itself can be used in place of an external transmission source **(7)**. Calculated correction factors can be verified using multiple gamma-ray energies from the nuclide in the item.

10.4.3 Methods for estimating the self-attenuation correction include the following:

10.4.3.1 Using measurement information to estimate holdup thickness coupled with computations involving published linear or mass attenuation coefficients.[6]

10.4.3.2 Perform transmission measurements using an external radiation source.

10.4.4 If the material matrix particle size is sufficiently small, for example, thin-film holdup, the self-attenuation correction may be negligible. This must be evaluated since the attenuation effect depends on the holdup material and its bulk density at the location.

10.5 *Assay Plan*—The assay plan should provide clear instructions defining the considerations affecting the quality of holdup measurements. These considerations might include instrumentation and support equipment, instrument settings, calibration and calibration checks, measurement locations, measurement distances, collimation and shielding, measurement times or accumulated counts, background measurement, and measurement control.

10.6 *Documentation*—The assay plan and the underlying assumptions and decisions should be documented.

11. Measurement Preparations

11.1 Measurement preparation consists of an evaluation of the facilities' measurement need, selection, and preparation of standards, and preparation of the measuring apparatus. Additional information can be found in ANSI N15.20.

11.2 *Preparation of Apparatus*—Prior to use the apparatus must be checked to assure its proper performance. Documentation of these specifications, the checks performed, and all adjustments required to bring instrumentation into specifications should be maintained with program quality assurance records and must meet facility and regulatory requirements.

11.3 *Standard Selection and Preparation*—Ideally, standards match the items to be measured with respect to isotopics, chemical form, geometry, containment, and SNM mass. This is rarely feasible. Standards must be selected or constructed carefully so they correctly support the selected holdup measurement method and model.

11.3.1 Differences between the geometry or containment of standards and those of the item to be measured must be addressed in the model used to interpret that data. The choice of model determines how many standards are needed. In some cases, a well-characterized point source standard will suffice to generate all the calibration constants needed **(8)**.[6]

11.3.2 If the measurement method and model use the item-specific approach, a standard or standard set, which closely matches the actual holdup distribution, will be required. Additionally, duplicate items will be needed to match the item attenuation in the calibration.

11.4 *Validation of the Calibration*—After establishment of a baseline for future measurement control by calibrating the measurement system, different approaches can be taken to validate the calibration.

11.4.1 *Holdup Removal*—When possible, a calibration may be verified by quantitatively removing the holdup and analyzing its nuclear material content by suitable destructive or nondestructive assay methods.

11.4.2 *Verification Using Standards*—In some cases, a standard can be placed in process equipment and measured. Care is needed to assure that the location of the standard within the process equipment simulates the actual holdup locations.

11.4.3 *Alternate Measurement Technique*—This technique might be possible using another gamma ray from the holdup, using neutron measurement techniques, or by other means. Agreement between alternate methods provides some verification of measurement validity; however, a careful evaluation of

[6] LANL training class.

TABLE 1 Gamma-Ray Emission Rate of 1 g of Isotope[A]

Isotope	Energy (keV)	Emission Rate (γ/s·g)
^{235}U	186	4.58×10^{4} [B]
^{238}U (^{234m}Pa in secular equilibrium with ^{238}U	1001	104[C]
^{238}Pu	153	5.94×10^{6} [D]
^{238}Pu	766	1.39×10^{5} [D]
^{239}Pu	129	1.44×10^{5} [D]
^{239}Pu	414	3.42×10^{4} [D]
^{239}Pu	375–450	9.8×10^{4} [E]
^{239}Pu	275–500	1.4×10^{5} [E]

[A] Values calculated based on data from the following sources:
[B] Helmer, R. G. and Reich, C. W., "Emission Probabilities and Energies of Gamma-Ray Transitions from the Decay of U-235," *Int. J. Appl. Radiat. Isot.*, Vol 35, pp. 783–786, (1984).
[C] Duchemin, B. and Coursol, N., Be, M. M., "The Reevaluation of Decay Data for the U-238 Chain," Nuclear Instruments and Methods in Physics Research, A339, pp. 146–150, (1994).
[D] Test Method C 1030.
[E] "Nuclear Data Sheets," Vol 66, pp. 887–891.

TABLE 2 Interfering Radiation

Isotope to be Measured	Energy Region (keV)	Detector System	Interfering Isotope	Interfering Gamma (keV)	Alternate System or Gamma (keV)
^{235}U	185.7	Scintillation detector system	^{99}Tc	0-300 Bremsstrahlung	Ge detector
^{235}U	185.7	Scintillation detector system	^{234}Pa (^{238}U daughter)	766-1001 Compton continuum	Background subtraction
^{235}U	185.7	Scintillation detector system	^{208}Tl (^{232}U daughter, ^{232}Th daughter)	238.6 and Compton continuum	Ge detector or 143.8 keV
^{239}Pu	413.7	Ge	^{237}U (^{241}Pu daughter)	415.9 (208-keV pileup)	129.3
^{239}Pu	375–450 or 275–500	Scintillation detector system	^{237}U	332	390–450 or Ge detector
^{239}Pu	375–450 or 275–500	Scintillation detector system	^{241}Am	several	Ge detector
^{239}Pu	413.7	Scintillation detector system, Ge	^{233}Pa (^{237}Np daughter)	415.8	129.3
^{239}Pu	375–450 or 275–500	Scintillation detector system	^{208}Tl	583.1 and 510.7 Compton continuum and 277.4	390–450, Background subtraction or Ge detector

the measurement bias for the methods should be performed.

11.5 *Initialize Measurement Control*—To ensure and document proper operation of the measurement instrumentation throughout the measurement period, measurement control practices are utilized. An evaluation program (using valid statistical techniques) should be established for the measurement control information. This program will provide an indication that the measurement process is or is not in control, and determination of a measurement bias. While this program provides information helpful in adjusting control limits, these measurements typically do not benefit from microscopic adjustments in the control parameters. The measurement control data should be evaluated using a valid statistical technique. Additional QA practices are found in ANSI/ASME NQA-1-1983 and Guide C 1009.

11.5.1 Four measurement control concepts can be used, the check-source, measurements with no items present, working source measurements, and precision checks. A summary of the measurement-control checks is given in Table 3. If the measurement control check response is outside the acceptable limits, it is recommended that measurements not proceed until the problem is solved. Locations measured since the last measurement control check, which was within limits, may need to be assayed again.

11.5.1.1 *Check-Source Measurements*—These measurements assure that the calibration of the measurement system has not changed. Sources are centered at a fixed distance from the detector face and measured for a fixed time. A check-source data set is established immediately following instrument calibration. For subsequent measurements, ranges of acceptable results (count rates) need to be established to assure that measurement equipment is in proper working order. Check-source measurements should be taken at the beginning and at the ending of the day (or shift), or more often, if significant instability is suspected due to temperature, humidity fluctuations, or other reasons.

TABLE 3 Measurement-Control Check Summary

Measurement-Control Check	Item(s) Checked
Check source	Measurement-system response, region of interest (energy window) adjustment
No item present	Detector contamination
Working source	Detector collimation, repeatability, region of interest (energy window) adjustment
Precision check	System repeatability

11.5.1.2 *Measurements With No Items Present*—Measurements should be conducted in a region with low and consistent gamma-ray background at a frequency established by the measurement control program. These measurements can help verify system stability and indicate detector contamination.

11.5.1.3 *Working Sources*—These sources typically a process equipment item, may be used to verify that instrument response has remained stable with time; to verify adherence to procedures, proper operation of measurement instrumentation, proper adjustment of the collimator, and consistency of other parts of the measurement program. They also are helpful for evaluating the uncertainty due to positioning of the equipment by measurement personnel. Depending on the use of the working source, knowledge of material quantities may or may not be required. A working source should contain the isotope of interest or use an isotope that reasonably matches the gamma-ray characteristics of the SNM to be measured. As well, the physical characteristics, for example, overall size, of the process equipment should be matched if feasible. Actual holdup can be used as the working source even if an accurate analytical value of the material present is not known.

11.5.1.4 *Precision Check*—Repeatability data that test for the significance of each of the above effects should be

developed at the outset and continued throughout the measurement program. Long-term repeatability can be assessed also by another program, which includes repeat measurements of a working source (or any static item) at regular intervals by all measurement personnel.

12. Assay

12.1 The initial procedure for measurement of an item can differ substantially from that used for subsequent measurements. Unless circumstances change sufficiently to require modification of procedures, subsequent measurements of an item can follow the procedures established from the previous analysis and assessment of results. Measurement control measurements are interspersed between measurements of unknown items. In addition the background must be assessed at the measured item.

12.2 Once the assay requirements have been determined and the measurement technique established, final preparations, and execution of assay measurements may commence. Holdup measurements may be intrusive to process operations and may require nuclear material transfers or clean out.

13. Calculation

13.1 Calculations are performed as appropriate to the chosen calibration model and measurement techniques **(9)**.

14. Precision and Bias

14.1 Due to the unique nature of holdup measurements, it is recommended that users develop precision and bias estimates for their own application of the measurement techniques described in this guide. Causes of uncertainties associated with holdup measurements fall into four broad categories:

14.1.1 Lack of information concerning the actual measurement geometry, the distribution of SNM, and the true attenuation of the measured signal;

14.1.2 Uncertainties resulting from use of overly simple models;

14.1.3 Uncertainties in evaluating the background, including counting statistics; and,

14.1.4 Counting statistics associated with the item measurement.

14.2 Of these four causes, counting statistics is easily controlled for all but the smallest holdup, causes the smallest contribution to overall measurement error, and in 14.1.4 is considered to be a source of random error. Of these four categories the lack of information causes the largest difficulties. The first three categories tend to cause biased results, though most holdup measurements yield no indication of the potential for bias.

14.3 *Precision*—The precision of holdup measurements varies widely from assay situation to assay situation. Three of the four categories above can affect measurement precision. Application of simple models is the lone exception. Specific factors that affect measurement precision include the following: counting statistics, detector positioning, instrumentation differences, operator dependent effects, and environmental effects. Some of these factors may combine to produce greater effects than the sum of the individual effects. Repeat measurements can provide data for estimating precision errors relating to many of the listed factors. Extensive work may be required to provide a statement of precision for an individual assay situation or location. If necessary, a plan to perform the required measurements to determine the precision can be developed and executed.

14.4 *Bias*—It is not practical to specify the bias of the techniques described in this guide since each assay location or situation, with few exceptions, is unique. Biases as large as 100 % have been reported. With the exception of counting statistics all of the categories above contain factors, which affect measurement bias. Factors that can significantly affect the bias include improper detector positioning, errors in estimation of attenuation corrections, nonuniformity of holdup material, incorrect modeling of process equipment, incorrect background subtraction (both peaked and Compton), plus incorrect assumptions regarding isotopic composition, incorrect model regarding process equipment physical factors, and gamma-ray interferences.

REFERENCES

(**1**) Reilly, D., Ensslin, N., Smith, Jr., H., Kreiner, S., *Passive Nondestructive Assay of Nuclear Materials*, U. S. Nuclear Regulatory Commission, Washington, DC, 1991, p. 601.

(**2**) Matussek, P., "Accurate Determination of ^{235}U Isotope Abundance by Gamma Spectroscopy," *A User's Manual for the Certified Reference Material EC-NRM-171/NBS-SRM-969*, KfK 3752, Kernforschungszentrum Karlsruhe GmbH, Karlsruhe, May 1985.

(**3**) R. S. Marshall, SNM Holdup Assessment of Los Alamos Exhaust Ducts, Final Report, LA-12700 (February 1994).

(**4**) R. R. Picard," Measurement Campaigns for Holdup Estimation," *Journal of Nuclear Materials Management*, Vol XVI, Number 4 (July 1988).

(**5**) Nondestructive Assay of Special Nuclear Materials Holdup," LANL Training Course Manual, February 1991.

(**6**) Parker, J. L. "The Use of Calibration Standards and the Correction for Sample Self-Attenuation in Gamma-Ray Nondestructive Assay," LA-10045, Revised 1986, LANL.

(**7**) Hagenauer, R. C., "Nondestructive Assay Quantification of Radioactive Isotopes in Poorly Characterized Containers," *Trans. Amer. Nuc. Soc. Annual*, Vol 76, pp. 124–125.

(**8**) K. E. Thomas, S. P. Pederson, N. R. Zack, S. A. Jones, and B. R. McGinnis, Holdup Data Analysis for Portsmouth Building X705, LA-UR-91-2468, presented at 32nd Annual Meeting of the INMM, New Orleans, USA (July 28–31, 1991).

(**9**) Detailed information on general calculations can be found in: Reilly, *et al*, ibid chapters 5 and 6, and C. H. Kindle, "In Situ Measurement of Residual Plutonium," ARH-SA-248. Examples of calculations for specific calibration models are given in: LANL Training Course Manual, *ibid*; G. A. Sheppard, P. A. Russo, T. R. Wenz, M. C. Miller, E. C. Piquette, F. X. Haas, J. B. Glick and A. G. Garrett, "Models for Gamma-Ray Holdup Measurements at Duct Contact," LA-UR-91-2505, 32 INMM Meeting, New Orleans (July 1991) and; Kindle, *ibid*.