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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 hel 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

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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.

the sample or item being measured, can be considered background. The three main contributors to background are as follows:

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.