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

This standard is issued under the fixed designation C 1455; 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 gamma-ray methods used to nondestructively measure the quantity of ^{235}U, or ^{239}Pu remaining as holdup in nuclear facilities. Holdup occurs in all facilities where nuclear material is processed, in process equipment, in exhaust ventilation systems and in building walls and floors.

1.2 This test method includes information useful for management, planning, selection of equipment, consideration of interferences, measurement program definition, and the utilization of resources (**1, 2, 3, 4**).[2]

1.3 The measurement of nuclear material hold up in process equipment requires a scientific knowledge of radiation sources and detectors, transmission of radiation, calibration, facility operations and error analysis. It is subject to the constraints of the facility, management, budget, and schedule; plus health and safety requirements; as well as the laws of physics. The measurement process includes defining measurement uncertainties and is sensitive to the form and distribution of the material, various backgrounds, and interferences. The work includes investigation of material distributions within a facility, which could include potentially large holdup surface areas. Nuclear material held up in pipes, ductwork, gloveboxes, and heavy equipment, is usually distributed in a diffuse and irregular manner. It is difficult to define the measurement geometry, to identify the form of the material, and to measure it without interference from adjacent sources of radiation.

1.4 *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:* [3]

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

C 1490 Guide for the Selection, Training and Qualification of Nondestructive Assay (NDA) Personnel

C 1673 Terminology of C26.10 Nondestructive Assay Methods

2.2 *ANSI Standards:*[4]

ANSI N15.20 Guide to Calibrating Nondestructive Assay Systems

2.3 *U.S. Nuclear Regulatory Commission Regulatory Guides:*[5]

Regulatory Guide 5.23, In Situ Assay of Plutonium Residual Holdup

3. Terminology

3.1 Refer to Terminology C 1673 for definitions used in this test method.

4. Summary of Test Method

4.1 *Introduction*—Holdup measurements range from the solitary 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 or items instead of on multiple items with similar characteristics measured in a specialized, isolated room. Often the chemical form and geometric distribution of the SNM are not well known. These challenges require unique preparation

[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.

Current edition approved June 1, 2007. Published July 2007. Originally approved in 2000. Last previous edition approved in 2000 as C 1455 – 00.

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

[3] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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

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

for every measurement to obtain a quality result but unknowns can lead to large measurement uncertainties.

4.2 *Definition of Requirements*—Definition of the holdup measurement requirements should include, as a minimum, the measurement objectives (that is, criticality control, SNM accountability, safety, or combinations thereof); time and resource constraints; the desired measurement sensitivity, accuracy, and uncertainty, and available resources (schedule, funds, and subject matter experts).

4.3 *Information Gathering and Initial Evaluation*—Information must be gathered concerning the item or items to be assayed and an initial evaluation should be made of the level of effort needed to meet the holdup measurement requirements. Preliminary measurements may be needed to assess the problem; to define the location and extent of the holdup, to determine the SNM isotopic composition or enrichment, and to identify potential interfering radionuclides. 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, facility processing status, radiological and industrial safety considerations, plus the personnel and equipment needed to complete the assay. Sources of information may include a visual survey, engineering drawings, process knowledge, process operators, and prior assay documentation.

4.3.1 Subsequent measurement campaigns may well proceed faster, especially when the objective is to quantify changes from the previous measurement campaigns and no changes have been made to the process.

4.3.2 Shutdown facilities are frequently measured once through carefully and completely. Any subsequent measurement campaigns may only verify a subset of the data set.

4.4 *Task Design and Preparation*—The initial evaluation provides a 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 will include measurement locations and geometries or guidance for their selection, it typically outlines required documentation, operating procedures, background measurement methods and frequencies, plus training, quality and measurement control requirements (Guide C 1009). Necessary procedures, including those for measurement control, should be developed, documented, and approved.

4.4.1 During the initial measurement campaign the task design and preparation may require 50% of the time allotted to the measurement campaign, subsequent campaigns typically require a much smaller fraction of the total measurement campaign time for task design and preparation.

4.5 *Calibration*—Calibration and initialization of measurement control is completed before measurements of unknowns. Calibration requires traceable standards.

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

4.7 *Evaluation of Measurement Data*—As appropriate, corrections are made for gamma-ray attenuation effects, for example, the container, item matrix, absorbers, and measured background. As appropriate, corrections are made for finite geometry effects. These corrections are applied in the calculation of the assay value. Measurement uncertainties are established based on factors affecting the assay.

4.7.1 Converting measurement data to estimates of the quantity of nuclear material holdup requires careful evaluation of the measurement parameters 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 (random and systematic) are estimated based on uncertainties in assay parameters, for example, holdup distribution, attenuation effects, measured count rates and finite source corrections.

4.7.2 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.

4.8 *Documentation*—Measurement documentation should include the plans and procedures, a description of measurement parameters considered important to the calibration and for each measurement location, the measurement techniques used, the raw data, assumptions and correction factors used in the analysis, the results with estimated precision and bias, and comparison to other measurement techniques.

5. Significance and Use

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

5.2 *Scan*—The scan is used to provide a qualitative description of the extent, location, and the relative quantity of holdup. It can be used to plan or supplement the quantitative measurements.

5.3 *Nuclide Mapping*—Nuclide mapping measures the relative isotopic composition of the holdup at specific locations. It can be used to detect the presence of radionuclides that emit radiation which could interfere with the assay. If the holdup is not isotopically homogeneous at the measurement location, that measured isotopic composition will not be a reliable estimate of the bulk isotopic composition.

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

5.4.1 High quality results require detailed knowledge of radiation sources and detectors, transmission of radiation, calibration, facility operations and error analysis. Judicious use of subject matter experts is invaluable (Guide C 1490).

5.5 *Spot Check and Verification Measurements*—Periodic re-measurement of holdup at a defined point using the same technique and assumptions can be used to detect or track relative changes in the holdup quantity at that point over time. Either a qualitative or a quantitative method can be used.

5.6 *Indirect Measurements*—Quantification of an radionuclide can be determined by measurement of a daughter radionuclide or of a second radionuclide if the ratio of the abundances of the two radionuclides is known. This can be used when there are interfering gamma rays or when the parent radionuclide 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 radionuclides be known with sufficient accuracy.

5.7 *Mathematical Modeling*—Modeling is an aid in the evaluation of complex measurement situations. Measurement data are used with a mathematical model describing the physical location of equipment and materials. (**3**, **5**, **6**, **7**, **8**).

6. Interferences

6.1 Background can cause problems in several ways. It may contribute undesired events to either the peak of interest or to the background continuum. Consequently it can cause a bias or have deleterious effects on the precision, or both.

6.1.1 Gamma-rays from the isotope being measured that do not originate in the item being measured can bias results high.

6.1.2 Background variations can cause biased results. For example, SNM in nearby items that are moved or shielding that is moved during the measurement can cause biased results.

6.1.3 If a background activity 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 overestimated, underestimated or missed altogether.

6.1.4 *Interfering gamma ray peaks*—Gamma-rays emitted by nuclides other than the nuclide of interest may produce a bias if the gamma ray energies are sufficiently close to each other. For example low resolution detectors do not easily distinguish the 185.7 keV gamma ray from ^{235}U from the 239.0 keV gamma ray from ^{212}Pb, or the 413.7 keV gamma ray from ^{239}Pu from the 662.0 keV gamma ray from ^{137}Cs.

7. Apparatus

7.1 General guidelines for selection of detectors and signal-processing electronics are discussed elsewhere.

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 will choose a suitable trade-off between detector energy resolution, detection efficiency, equipment complexity and equipment portability (weight, size and number of pieces).

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 (BGO) or NaI detector. The detection system may be as complex as a Ge-detector with a complete 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.

7.5 *Medium Resolution Measurement Systems*—CdZnTe or $LaBr_3$ are newer, medium resolution gamma-ray detectors. Resolution is typically adequate to obtain isotopic information from the spectra.

7.6 *High Resolution Measurement Systems*—A high resolution gamma-ray spectrometry system may be necessary if the isotopic distribution varies or interfering gamma-rays are present. 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, but weigh more and require more care and attention.

7.7 *Detector Collimation and Shielding*:

7.7.1 A collimator is often 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.

7.7.1.1 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.

7.7.1.2 Changes in the absorber foils or detector field of view causing a change in the calibration will require a change in the response model of the detector system whether it is determined empirically or calculated.

7.7.2 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.

7.7.3 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, absorber foils can be used to reduce high count rates, which can produce spectral distortions and biases in the assay results.

7.8 *Detector Positioning Apparatus*—such as measuring and pointing devices or support stands to help attain reproducible geometry 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 in 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.

8.1.5 Some holdup detectors require liquid nitrogen; proper industrial safety practices must be followed.

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 can significantly alter assay results. For example, noise or microphonics can degrade the energy resolution of the spectra.

8.2.3 *Secular Equilibrium*—If the gamma ray from a daughter radionuclide is used to quantify holdup, such as with ^{238}U and ^{234}mPa, secular equilibrium within the holdup should be verified. If secular equilibrium is assumed but not established measurement results could be biased.

8.2.4 *Infinitely Thick SNM Holdup*—If the holdup deposit is infinitely thick to the measurement of gamma rays, transmission corrections are not simple to perform and the measurement results will likely be biased low.

8.2.5 *Background*—lack of understanding of background effects on the measurement or incorrect background measurements may impact the results significantly. Measurement items or items affecting background should not be moved during measurements.

8.2.5.1 It can be challenging to position the detector to properly account for background.

8.2.6 Temperature changes at the measurement location may result in a detector gain drift. Stabilization methods may be necessary to mitigate this effect.

8.2.7 Unexpected presence of brehmstralung in the spectra may cause a bias in low resolution measurements. For example, brehmstralung caused by ^{99}Tc or ^{238}U.

9. Procedure

9.1 A Holdup Measurement Campaign procedure generally includes the following:

9.1.1 Development (or Review) of Measurement Strategy and Development (or Review) of Detailed Measurement Plan,

9.1.2 Preparation for Measurements,

9.1.3 Perform the Measurements,

9.1.4 Calculations (often in parallel while the data is acquired),

9.1.5 Estimation of Measurement Uncertainty (typically Precision and Bias), and,

9.1.6 Recording of data and results (**3**, **4**, **9**, **10**, **11**) NRC Regulatory Guide 5.23).

9.2 *Procedure—Measurement Strategy/Plan Development*:

9.2.1 *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:

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

9.2.1.2 Radionuclide or radionuclides of interest.

9.2.1.3 Acceptable level of measurement uncertainty.

9.2.1.4 Acceptable lower detection limit for the assay.

9.2.1.5 Intended and potential applications for results, for example, criticality risk assessment, SNM accountability, health physics, or decontamination and demolition.

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

9.2.2 *Constraints that are useful to know about:*

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

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

9.2.3 *Personnel and Procedures*—Note there are typically two levels of procedures: generic or all encompassing such as the measurement strategy or selection of models and the detailed work instructions for each data acquisition:

9.2.3.1 Since holdup measurements are made with little or no sample preparation and under a wide range of conditions, formal procedures might be developed for the item measurements. Procedures can evolve to incorporate lessons learned from previous experience.

9.2.3.2 Personnel performing holdup measurements must have adequate training, education, and experience. Definition of adequate training, educations, and experience can be found in Guide C 1490. Development of measurement plans, strategy and work instructions and the initial measurements generally require much more expertise than routine or subsequent re-measurements, which can be performed by trained personnel using established procedures.

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

9.2.5 *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.

9.2.5.1 Inspect the area(s), equipment, or both, 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.

9.2.5.2 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 important insights for consideration. 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.

9.2.5.3 Obtain accurate engineering drawings, if they are available. The drawings are useful during the identification of measurement locations, determination of physical measurement techniques and development of attenuation corrections.

9.2.5.4 Obtain information such as the process flow sheets 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 detectable movement of SNM during measurements of process components.

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

9.2.5.6 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 can be noted for later quantitative measurements.

9.2.5.7 Removal of background sources, attenuating equipment, and extraneous items can facilitate subsequent measurements, requiring less time and resources and providing more accurate results.

9.3 *Procedure—Develop Detailed Measurement Plan*—A critical step in the evaluation process is the determination of how the measurements will be performed. For most facilities, a generalized geometry model can provide acceptable results for most items using the least amount of resources (**3**). However, nearly all facilities will also have special cases that require specialized models (**5**, **6**, **8**, **11**).

9.3.1 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 (**9**, **10**).

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

9.3.3 Measurements of an item at multiple distances or from different directions, when possible, can sometimes provide reassurance assumptions are consistent with the measurement results.

9.3.4 Measurements made at a distance from the item are less sensitive to how the SNM is distributed than measurements made close to the item. Interferences, neighboring background items, 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.

9.3.5 *Selection of Measurement Techniques*—Other factors that are generally determined 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, and distance between contiguous measurements.

9.3.6 *Attenuation Correction*—Estimates of attenuation correction factors for the container wall, the material matrix (self-attenuation), and the effects of lumps must be determined. Some available methods for estimating attenuation corrections are:

9.3.6.1 Measurements, and published linear or mass attenuation coefficients (**12**).

9.3.6.2 Measure the transmission using an external radiation source (**3**, **5**).

9.3.6.3 Multiple gamma-ray energies from the nuclide in the sample itself have been used in place of or in conjunction with an external transmission source (**5**, **7**). Calculated correction factors can be assessed using analysis based on different gamma-ray energies from the radionuclide in the item.

9.3.6.4 If the material matrix particle size and thickness in the direction of measurement is sufficiently small, the self-attenuation correction may be negligible.

9.3.7 *Assay Plan*—The assay plan should provide clear instructions regarding everything affecting the quality of the holdup measurements. These considerations include support equipment, instrument settings, calibration and calibration checks, measurement locations, measurement distances, collimation and shielding, measurement times, background measurement, and measurement control (Guide C 1009).

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

9.4 *Procedure—Preparations for the Measurements*:

9.4.1 Measurement preparation consists of selection and preparation of standards, and preparation of the measuring apparatus. Additional information can be found in ANSI N15.20.

9.4.2 *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 quality assurance records and must meet facility and regulatory requirements.

9.4.3 *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 for holdup measurements, typically one must rely on simple point sources. Standards should be selected or constructed carefully so they correctly support the selected holdup measurement method and model.

9.4.3.1 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 (**3**, **6**, **11**).

9.4.3.2 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, the standards will need to match the item attenuation (**5**, **11**).

9.4.4 *Validation of the Calibration*—Different approaches can be taken to validate the calibration.

9.4.4.1 *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.

9.4.4.2 *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.

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

9.4.5 *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. The measurement control data should be evaluated using a valid statistical technique (Guide C 1009).

9.4.5.1 Three measurement control concepts can be used, the check-source, measurements with no items present, and working source measurements. A summary of the measurement-control checks is given in Table 1. 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.

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 end of the measurement day (or shift). If significant instability is suspected due to temperature, humidity fluctuations, or other reasons, additional measurements should be made.

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.

Working Sources—These sources, often a contaminated 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 radionuclide of interest or use an radionuclide 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.

9.4.5.2 Precision checks or repeatability evaluations, if desired, are generally done with working sources or process items.

9.5 *Perform the Measurement*:

9.5.1 The initial measurement of an item typically requires the most time for preparation of measurement strategy, work instructions, and the actual measurement.

9.5.1.1 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.

9.5.2 The background is best assessed at the measured item, since background levels can vary widely around the measurement locations. Sometimes several measurements are useful in identifying the background sources potentially affecting the measurement.

TABLE 1 Gamma-Ray Emission Rate of 1 g of Isotope

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

9.5.2.1 The simplest approach to measuring background at a holdup measurement location is often to aim the detector next to the item being measured or at a point behind the item being measured.

9.5.2.2 If this is not convenient, shadow shielding might be useful in reducing the intensity of a background source or the intensity of the item being measured, thereby facilitating the background measurement.

9.5.2.3 Plugs made of high-Z materials that fit snugly in the detector collimator opening can be used to block the signal from the measurement item, allowing a measurement of the background coming from behind and from beside the detector to be made.

9.5.3 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.

9.6 *Procedure—Calculations*—The documentation for the calculations should include what was done, the steps followed, assumptions, and any necessary justification.

9.6.1 Calculations are performed as appropriate to the chosen calibration model and measurement techniques (**6**). An illustrative example is the generalized geometry holdup (GGH) approach that models the measurement items as points line, or area sources. GGH was developed, in part, to accommodate the need for calibrating with small point sources when representative standards were not available. By examining the facility and judiciously approximating the measurement geometry at each location, one of only three distinct models can be used to assay holdup with generally acceptable accuracy at each of hundreds of unique locations (**3**).

9.6.2 GGH may not always meet the user's needs. Other approaches are generally not as fast as the GGH because they include specific features such as geometry or attenuation or calibration modeling that is specific to each measurement location. By investing more time in set up and modeling, the user can often obtain more accurate results (**3**, **5**, **6**, **8**).

9.7 *Procedure - Estimate Precision and Bias*—Due to the measurement location specific 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 test method. While in general, the quality of the results improves with increased level of effort, it is important for the user to not invest time and money in attempting to improve estimating measurement uncertainties beyond the point of diminishing returns. Holdup measurement uncertainties are generally larger than those for other measurements. Possible values for comparing such estimates can be found in reference (**13**).

10. Precision and Bias

10.1 Causes of uncertainties associated with holdup measurements fall into four broad categories:

10.1.1 Lack of information concerning the actual measurement item (including the geometry of the holdup), the distribution and type of SNM, and the true attenuation of the measured signal can cause bias as large as a factor of 6 (600%);

10.1.2 Uncertainties resulting from use of overly simple models can cause bias of 10-25%;

10.1.3 Uncertainties in evaluating the background, have caused bias as large as a factor of 3; and,

10.1.4 Counting statistics associated with the item measurement generally impact the precision of the result and can be most easily addressed.

10.1.5 Of these four causes, counting statistics is easily controlled for all but the smallest holdup, causes the smallest contribution to overall measurement error, and is considered to be a source of random error. Of these four categories the lack of information about the measurement geometries generally causes the largest difficulties. The first three categories tend to cause biased results, though most holdup measurements yield little or no indication of the potential for bias. While biases can occur in both directions, in most situations with bias, the holdup measurement results are biased low (**13**).

10.2 Each facility (or building or process) should use results from their own cleanout and recovery to validate the precision and bias estimates.

10.3 *Precision*—The precision of holdup measurements varies widely from assay situation to assay situation. Specific factors that affect measurement precision include the following: counting statistics, detector positioning, instrumentation differences, human error, and environmental effects.

10.3.1 Some of these factors may combine to produce greater effects than the sum of the individual effects.

10.3.2 Repeat measurements can provide data for estimating precision errors.

10.3.3 Longer counting times can reduce the effects of some of the listed factors on measurement precision.

10.3.4 Automation (including careful documentation) has been shown to improve measurement precision.

10.4 *Bias*—It is not practical to succinctly specify the bias of the techniques described in this test method since each assay location or situation, with few exceptions, is unique. Biases as large as 244 % have been reported (**13**). High quality cleanout data has been shown to be useful in improving the measurements and the analysis. All of the factors mentioned previously can affect measurement bias. Additional factors include nonuniformity of the deposit, errors in estimation of corrections, incorrect modeling, incorrect background subtraction, plus incorrect assumptions regarding isotopic composition and gamma-ray interferences.

10.4.1 Unfortunately holdup measurement bias factors may not be independent or symmetric. Combining them in quadrature may not be the best approach. Sometimes summing some of the bias factors is statistically defensible.

10.4.2 The following are measurement biases reported by subject matter experts (Guide C 1490) using low resolution equipment, the generalized geometry model, and cleanout results (measured result/reference value):

10.4.2.1 Measurements of HEU processing can vary from 14% to 137%.

10.4.2.2 Measurements of LEU processing can vary from 25% to 244%.

10.4.2.3 Measurements of Pu processing can vary from 10% to 157%.

10.4.2.4 After adjusting the calculational models based on the cleanout values, the measured and reference values can agree as well as 10-20%. More details are in references (**13 and 14**).

10.4.3 Experience indicates previous results or results from other process areas or buildings or facilities may not be reliable indicators of the bias in subsequent holdup measurements.

10.4.4 In most situations, if the holdup result is biased, the measurement is low compared to the actual value.

11. Keywords

11.1 holdup,; holdup measurements; in-process inventory; material holdup; nuclear material holdup

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