Designation: C1455 − 14 (Reapproved 2023)

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 C1455; 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 present as holdup in nuclear facilities. Holdup may occur in any facility 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 uncertainty analysis. It is subject to the constraints of the facility, management, budget, and schedule; plus health and safety requirements. 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

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

C1592 Guide for Making Quality Nondestructive Assay Measurements

C1673 Terminology of C26.10 Nondestructive Assay Methods

2.2 *ANSI Standards:*[4]

ANSI N15.36 Measurement Control Program—Nondestructive Assay Measurement Control and Assurance Systems

ANSI N15.56 Nondestructive Assay Measurements of Nuclear Material Holdup: General Provisions

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 C1673 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 or routine measurement of a piece of equipment, to an extensive campaign of determining the total SNM in-process inventory for a processing plant.

[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 Jan. 1, 2023. Published January 2023. Originally approved in 2000. Last previous edition approved in 2014 as C1455 – 14$^{\varepsilon 1}$. DOI: 10.1520/C1455-14R23.

[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.

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 for every measurement to obtain a quality result. Unknown measurement parameters 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); the desired measurement sensitivity, measurement uncertainty, and available resources (schedule, funds, and subject matter experts). The customer, the measurement organization, and appropriate regulatory authorities should agree on the holdup measurement requirements before holdup measurements commence.

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 more rapidly 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. The measurement plan will reference overall measurement program documents governing required documentation, operating procedures, background measurement methods and frequencies, plus training, quality and measurement control requirements. Any needed additional procedures should be developed, documented, and approved.

4.5 *Calibration*—Calibration and initialization of measurement control should be 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 to measured count rates are made for Compton background, gamma-ray attenuation effects by equipment walls, and measured area background. As appropriate, corrections are made for finite geometry effects in the assay model and for self-attenuation. 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, if 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 measurement location, the measurement techniques used, the raw data, the assumptions and correction factors used in the analysis, the results with estimated uncertainty, and comparison to other measurement techniques (when available).

5. Significance and Use

5.1 Measurement results from 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. This method 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*—A scan is used to provide a qualitative indication 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 also be used to detect the presence of radionuclides that emit radiation which could interfere with the assay. Nuclide mapping is best performed using a high resolution detector (such as HPGe) for best nuclide and interference detection. 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 the measured nuclides 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 required (Guide C1490).

5.5 *Holdup Monitoring*—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*—Quantity of a 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 and secular equilibrium (Terminology C1673) is present. 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 to meet assay uncertainty goals.

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 Not properly accounting for background can cause problems in several ways. Background may contribute undesired events to either the peak of interest or to the background continuum underneath the peak of interest. Consequently it can cause a bias, 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 background gamma-ray flux 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 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 considering the resolution of the measurement system. For example, low resolution detectors do not easily distinguish the 375.0 keV and 413.7 keV ^{239}Pu gamma rays from ^{237}Np interferences at 312.9 keV, 340.8 keV, 398.6 keV, and 415.8 keV. Plutonium holdup measurements using the 330-414 keV region also have age-dependent interferences from ^{241}Am in that region which are considerable for low resolution detectors.

7. Apparatus

7.1 General guidelines for selection of detectors and signal-processing electronics are discussed below (see Guide C1592).

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 may be 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).

7.2.1 *Scan Measurement Systems*—The minimum gross gamma-ray detection system may be a survey meter. If limited energy discrimination is satisfactory, 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.

7.2.2 *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.

7.2.3 *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.

7.2.4 *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. Germanium detector systems usually weigh more and require more care and attention than scintillator-based systems.

7.2.5 *Detector Collimation and Shielding:*

7.2.5.1 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.

7.2.5.2 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 detector is intended to be used and calibrated with a specific collimator, it is appropriate to refer to the unit as a detector-collimator assembly.

7.2.5.3 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.

7.2.5.4 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.

7.2.5.5 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 from ^{241}Am and Pu x-rays, which can produce spectral distortions and biases in the assay results.

7.2.6 *Detector Positioning Apparatus*—Mechanical apparatus to hold, position, and point the detector at holdup deposits is necessary to attain reproducible measurements under severe measurement geometry constraints.

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 is 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 for working with cryogenic liquids 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. Dead time indication from the measurement electronics will often identify this problem. Preliminary scan measurements (5.2) may also identify this problem.

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

8.2.3 *Secular Equilibrium (Terminology C1673)*—If the gamma ray from a daughter radionuclide is used to quantify holdup, such as with ^{238}U and ^{234m}Pa, secular equilibrium within the holdup should be verified. Process knowledge and history may provide the necessary information to determine if secular equilibrium has been established. If secular equilibrium is assumed but not established measurement results could be biased.

8.2.4 *Infinitely Thick (Terminology C1673) 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.

8.2.4.1 Reference (**3**) provides a detailed discussion on the corrections for thick deposits and the limitations of such corrections. The discussion in reference (**3**) applies directly to the GGH method although the principles discussed are applicable to all measurements.

8.2.5 *Background*—A lack of understanding of background effects on the measurement or incorrect background measurements may impact the results significantly. Neither measurement items nor items affecting background should be moved during measurements.

8.2.5.1 Care must be taken 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 bremsstrahlung in the spectra may cause a bias in low resolution measurements. For example, bremsstrahlung caused by ^{99}Tc or the ^{238}U daughter, ^{234m}Pa.

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 Calibration or Model Development,

9.1.4 Performance of Measurements,

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

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

9.1.7 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 a holdup measurement or campaign, 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(s) or piece(s) 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 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 *Resource Constraints:*

9.2.2.1 The time available to perform the measurement(s), analyze the data and report the results.

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

9.2.3 *Personnel and Procedures*—There are typically two levels of procedures: (*1*) generic or all-encompassing such as the measurement strategy or selection of models, and (*2*) the detailed work instructions for each data acquisition:

9.2.3.1 Formal procedures may be developed for the item measurements. Procedures can evolve to incorporate lessons learned from previous experience.

9.2.3.2 Personnel designing and performing holdup measurements must have adequate training, education, and experience. Definition of adequate training, educations, and experience can be found in Guide C1490. Development of measurement plans, strategy and work instructions and performing the initial measurements generally require much more expertise than the repeating of routine or subsequent remeasurements. Routine or subsequent remeasurements can be performed by trained personnel using established procedures and software.

9.2.4 *Safety Conditions*—Evaluation and mitigation 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. 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 equipment to be assayed and the surrounding area 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 Prior to the measurement campaign interview any personnel who may be familiar with the area(s) or equipment to be assayed. They may be able to provide first-hand information on current and historical process conditions, 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 measurements 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, reduce measurement time and resources and provide 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.

9.3.1 Several measurement techniques may be used.Select a measurement technique to be used. 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**). 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 *Instrument Selection*—Select a detector suitable for the measurements and identify the assay gamma ray(s) or band of energies.

9.3.3 Select a suitable assay calibration model considering 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.

9.3.4 Measurements of an item at multiple distances or from different directions can sometimes provide reassurance that assumptions are consistent with the measurement results.

9.3.5 *Select a suitable source to detector standoff.* Measurements made at a greater 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.

9.3.6 *Selection of Measurement Parameters*—Other parameters that must be selected for holdup measurements are count time, measurement locations, and distance between contiguous measurements.

9.3.7 *Physical Dimensions*—Depending on the assay calibration model selected, some physical dimensions of the