Designation: C1807 – 15

Standard Guide for Nondestructive Assay of Special Nuclear Material (SNM) Holdup Using Passive Neutron Measurement Methods[1]

This standard is issued under the fixed designation C1807; 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 guide describes passive neutron measurement methods used to nondestructively estimate the amount of neutron-emitting special nuclear material compounds remaining as holdup in nuclear facilities. Holdup occurs in all facilities in which nuclear material is processed. Material may exist, for example, in process equipment, in exhaust ventilation systems, and in building walls and floors.

1.1.1 The most frequent uses of passive neutron holdup techniques are for the measurement of uranium or plutonium deposits in processing facilities.

1.2 This guide includes information useful for management, planning, selection of equipment, consideration of interferences, measurement program definition, and the utilization of resources.

1.3 Counting modes include both singles (totals) or gross counting and neutron coincidence techniques.

1.3.1 Neutron holdup measurements of uranium are typically performed on neutrons emitted during (α, n) reactions and spontaneous fission using singles (totals) or gross counting. While the method does not preclude measurement using coincidence or multiplicity counting for uranium, measurement efficiency is generally not sufficient to permit assays in reasonable counting times.

1.3.2 For measurement of plutonium in gloveboxes, installed measurement equipment may provide sufficient efficiency for performing counting using neutron coincidence techniques in reasonable counting times.

1.4 The measurement of nuclear material holdup in process equipment requires a scientific knowledge of radiation sources and detectors, radiation transport, modeling methods, calibration, facility operations, and uncertainty 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. This guide does not purport to instruct the NDA practitioner on these principles.

1.5 The measurement process includes defining measurement uncertainties and is sensitive to the chemical composition, isotopic composition, 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, identify the form of the material, and measure it.

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

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

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C1009 Guide for Establishing and Maintaining a Quality Assurance Program for Analytical Laboratories Within the Nuclear Industry

C1455 Test Method for Nondestructive Assay of Special Nuclear Material Holdup Using Gamma-Ray Spectroscopic Methods

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

C1592/C1592M Guide for Making Quality Nondestructive Assay Measurements

C1673 Terminology of C26.10 Nondestructive Assay Methods

2.2 *NRC Standard:*

NRC Regulatory Guide 5.23 In-Situ Assay of Plutonium Residual Holdup[3]

[1] This guide 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, 2015. Published January 2015. DOI: 10.1520/C1807-15.

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

[3] Available from U. S. Nuclear Regulatory Commission (NRC), One White Flint North, 11555 Rockville Pk., Rockville, MD 20852-2738, http://www.nrc.gov.

2.3 *ANSI Standard:*
ANSI N15.20 Guide to Calibrating Nondestructive Assay Systems[4]

3. Terminology

3.1 *Definitions*—Refer to Terminology C1673 for definitions used in this guide.

4. Summary of Guide

4.1 *Introduction*—Holdup measurements using neutron methods typically measure the (α, n) or spontaneous fission production of neutrons, or both. Neutrons generated in items that do not include significant masses of neutron moderators, such as hydrogenous materials, typically have an escape fraction of nearly one. The isotopic distribution and, for (α, n) production, the chemical composition of the measured material affect assay results and shall be determined by process knowledge or an alternative measurement technique. Ref **(1)**[5] provides an example of a holdup campaign using neutron measurements.

4.2 *Choice of Measurement Method*—Passive neutron measurement methods are typically used for holdup when other methods of measurement (for example, gamma-ray assay) are not practical or would produce large biases. In some cases, neutron measurements are performed in conjunction with gamma-ray measurements for defense in depth or to obtain isotopic information, or both. Neutron measurement instrumentation is typically heavier, more difficult to shield, and has more difficult data interpretation than other NDA measurement methods. Neutrons, though, are very penetrating and less influenced by lumps than gamma rays, and the instrumentation has a very stable response. Examples of when neutron measurements are preferred include containers that severely attenuate gamma rays of interest for the nuclides measured or when sufficient nuclear material is present that self-attenuation of gamma rays of interest is severe (see Test Method C1455 and Guide C1592/C1592M).

4.3 *Specific Neutron Yield*—The number of neutrons generated per unit time per unit mass of the nuclide(s) of interest is an important parameter that is affected by conditions (for example, chemical composition and isotopic distribution) not detectable by passive neutron holdup measurement methods. Information used to estimate specific neutron yield shall be determined using process knowledge or alternate analysis methods (for example, sampling and X-ray fluorescence to determine chemical composition and high-resolution gamma-ray spectroscopy to determine isotopic composition). Both the chemical and isotopic distribution have significant effects on specific neutron yield.

4.4 *Definition of Requirements*—Definition of the holdup measurement requirements should include, as a minimum, the measurement objectives (that is, nuclear criticality safety, special nuclear material (SNM) accountability, radiological safety, or combinations thereof); time and resource constraints; the desired measurement sensitivity, accuracy, and uncertainty; and available resources (schedule, funds, and subject matter experts). Specific data quality objectives should be provided when available.

4.5 *Information Gathering and Initial Evaluation*—Information shall be gathered concerning the item or items to be assayed, and an initial evaluation should be made of the measurement techniques and level of effort needed to meet the holdup measurement requirements. Preliminary radiation measurements may be needed to define the location and extent of the holdup. Additional information should be collected prior to commencement of measurements. This information includes, but is not limited to, the geometric configuration of the item or process equipment to be assayed, location of the equipment in the facility, the presence of neutron moderators and absorbers, neutron leakage multiplication, factors affecting specific neutron yield, 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, results of sampling and wet chemical analysis, and prior assay documentation.

4.6 *Measurement Plan*—A measurement plan shall be developed. The initial evaluation provides a basis for choosing the quantitative method and assay model and, subsequently, leads to the determination of the detection system and calibration method to be used. Appropriate reference materials and support equipment are developed or assembled for the specific measurement technique. The plan will include measurement locations and geometries or guidance for their selection. In the plan, required documentation; operating procedures; background measurement methods and frequencies; plus training, quality, and measurement control requirements (Guide C1009) are typically outlined. Necessary procedures, including those for measurement control, shall be developed, documented, and approved.

4.7 *Calibration*—Calibration and initialization of measurement control is completed before measurements of unknowns. Calibration requires reference materials traceable to a National Measurement Institute to establish detection efficiency and modeling detector response to neutron sources. If modeling is used for calibration (for example, Monte Carlo n-Partical (MCNP) modeling), detailed specifications for the detector package will be required. If modeling is used, validation of the calibration shall include validation of each model developed. Familiarity with the facility on which assays will be performed is required to ensure that calibration is sufficiently robust to encompass all reasonable measurement situations.

4.7.1 *Calibration Using ^{252}Cf*—^{252}Cf is commonly used for calibrating neutron detectors. ^{252}Cf is convenient in that it provides a point source of neutron emissions with a strong signal so that calibrations can be completed using relatively short measurement times. Corrections for the difference in detection efficiency between neutrons from ^{252}Cf and neutrons from assayed items may be significant because of the difference in average energy from the two sources. For example, the

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

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

average energy of neutrons from ^{252}Cf is 2.14 MeV and the average energy of neutrons from holdup is 1.2 MeV for (α, n) with Fluorine as a target and an alpha energy of 5.2 MeV (**2**). An additional issue is that ^{252}Cf standards are typically certified for total neutron activity, and isotopes present in the standards produce an increasing number of neutrons as the mass of ^{252}Cf decreases relative to the mass of longer-lived isotopes as time passes. As the time since separation of the ^{252}Cf increases, this may become a significant source of bias unless appropriate corrections are made.

4.7.2 *Calibration Using Surrogate Materials*—Surrogate materials, typically created using the same materials that will be subsequently measured, may also be used for calibration, provided sufficient characterization is performed to establish traceability. These sources typically produce fewer neutrons per unit time than ^{252}Cf and require longer measurement times for equivalent calibration uncertainty. In addition, surrogate materials are typically significantly larger than point sources, which may complicate the process of evaluating calibration data. Calibration using surrogate materials reduces the number of corrections (for example, for energy difference between neutrons produced by the calibration source and measured materials) and may result in a lower total measurement uncertainty.

4.7.3 *Calibration Confirmation*—A calibration confirmation is needed to produce objective evidence demonstrating the applicability and correctness of the calibration relative to the items in which holdup is to be measured. The recommended method is to assemble test item(s) consisting of source/matrix and radioactive material configuration(s) nominally representative of the items to be characterized. The test item(s) should contain known and, preferably, traceable quantity of radioactive material in a known and representative configuration. If practical, the range of expected materials should be spanned. Acceptance criteria for the calibration confirmation measurements should be established in the measurement plan.

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

4.9 *Evaluation of Measurement Data*—As appropriate, corrections are estimated and made for factors that may bias the measurement. Examples include neutron scattering; cosmic ray induced spallation; leakage multiplication; neutron moderators, absorbers, and poisons; and the presence of targets that produce (α, n) neutrons. These corrections are applied in the calculation of the assay value. Measurement uncertainties are established based on factors affecting the assay.

4.9.1 Converting measurement data to estimates of the quantity of nuclear material holdup requires careful evaluation of the measurement parameters against calibration and modeling assumptions. Depending on the calibration, models, and measurement methods used, corrections may be necessary for geometric effects (differences between holdup measurement and calibration geometries); neutron moderators, absorbers, or poisons; scattering from nearby process equipment; the influence (scattering and shielding) of and holdup in nearby process equipment that is in the detector field of view; background; and interferences. Measurement uncertainties (random and item-specific bias) are estimated based on uncertainties in assay parameters. A comprehensive total measurement uncertainty analysis must accompany every measurement result.

4.9.2 Results should be evaluated against previous results or clean-out data, if either are available. This evaluation provides a cross-check between measurement techniques. The results of this evaluation can be used to provide feedback to measurement personnel, to refine the measurement and analysis techniques, and to evaluate the measurement uncertainty against estimates. If a discrepancy is evident, an evaluation should be made. Modeling errors or other sources of bias can be identified using this technique. Additional measurements with subsequent evaluation may be required. This can be used as a step in a phased approach.

4.9.3 If practical, measurements should be made of clean process equipment or, ideally, a plant that has not yet had nuclear material introduced. This provides a baseline for future measurement of holdup.

4.10 *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, a thorough description of the models used, the results with estimated precision and bias, and comparison to other measurement techniques when available.

5. Significance and Use

5.1 This guide assists in satisfying requirements in such areas as safeguards, SNM inventory control, nuclear criticality safety, waste disposal, and decontamination and decommissioning (D&D). This guide can apply to the measurement of holdup in process equipment or discrete items whose neutron production properties may be measured or estimated. These methods may meet target accuracy for items with complex distributions of SNM in the presence of moderators, absorbers, and neutron poisons; however, the results are subject to larger measurement uncertainties than measurements of less complex items.

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

5.2.1 High-quality results require detailed knowledge of radiation sources and detectors, radiation transport, calibration, facility operations, and error analysis. Consultation with qualified NDA personnel is recommended (Guide C1490).

5.2.2 Holdup estimates for a single piece of process equipment or piping often include some compilation of multiple measurements. The holdup estimate must appropriately combine the results of each individual measurement. In addition, uncertainty estimates for each individual measurement must be made and appropriately combined.

5.3 *Scan*—Radiation scanning, typically gamma, may be 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 neutron measurements. Other indicators (for example, visual) may also indicate a need for a holdup measurement.

5.4 *Nuclide Mapping*—To appropriately interpret the neutron data, the specific neutron yield is needed. Isotopic measurements to determine the relative isotopic composition of the holdup at specific locations may be required, depending on the facility.

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 quantitative method can be used.

5.6 *Indirect Measurements*—Neutron measurements do not identify the radionuclide that produced the neutron signal. The specific neutron yield shall be determined independently of the neutron measurement.

5.7 *Modeling*—Modeling is recommended as an aid in the evaluation of complex measurement situations. Measurement data are used with a radiation transport model that includes a description of the physical location of equipment and materials. Because of the complexity of neutron transport calculations, models are often developed using a transport code such as MCNP. Geometric models can also be used but, generally, do not account for phenomena such as scattering and estimation of neutron escape fraction.

6. Interferences

6.1 Background can cause a bias or have adverse effects on the precision or both. Because of scattering and physical limitations (for example, weight of the shielded detector package), background often cannot effectively be reduced to an insignificant level and is a significant contributor to total measurement uncertainty.

6.1.1 If background changes with measurement position, it may be necessary to develop a model of the background that incorporates the effects of measurement parameters (for example, physical structure, presence of concrete, personnel, height at which background is measured).

6.1.2 Unrecognized and uncompensated background variations can cause biased results. For example, SNM in nearby items that are moved or shielding that is moved during or between the commencement of the background and the completion of the assay measurement can cause biased results. This bias depends on the signal to background ratio.

6.1.3 Neutron production rates are often low and background rates are often large relative to the neutron flux from the holdup. In this case, the overall assay sensitivity will be reduced and uncertainty increased. Background measurements should be performed in a manner and at a frequency consistent with the signal-to-noise ratio for the desired measurement sensitivity.

6.1.4 *Interfering Neutron Production*—Neutrons generated by unexpected sources may produce a bias.

6.1.4.1 *Alpha Targets*—In assays of material for which (α, n) production is significant, specific neutron yield is proportional to the type and concentration of alpha targets. Incorrect assumptions of chemical composition and the presence of alpha targets (for example, impurities such as Be or O) that are not included in the model will result in a bias.

6.1.4.2 *Neutron-Producing Radionuclides*—Neutron-producing radionuclides whose presence are not considered in the estimate of specific neutron yield will result in a bias.

6.1.4.3 *Gamma-ray Producing Radionuclides*—Gamma-rays can register as counts in neutron instrumentation. This effect is noticeable in non-^{3}He neutron detection systems, and in ^{3}He detection systems if the high voltage is set to a high value.

6.1.5 *Cosmic-Ray-Induced Spallation*—Neutrons produced by interaction between cosmic-ray spallation showers and both non-nuclear and nuclear materials in measured items (for example, steel container walls, lead included as part of the container wall or as a gamma-ray shield, and uranium) can cause a bias. Cosmic-ray induced spallation can result in a significant bias. Estimation of the cosmic-ray background should be performed close in time to the assay. Cosmic ray spallation can shift with changing conditions, such as atmospheric pressure and rain.

6.1.6 *Matrix Effects*—Matrix materials that are unexpected or improperly modeled can cause a bias. Examples include modeling for leaded glass when acrylic is substituted, modeling a full tank and then measuring an empty tank, and not accounting for the presence of Raschig rings. Peer review of calculations and measurement assumptions can be used to limit this type of bias.

7. Apparatus

7.1 The apparatus chosen for measurements shall have capabilities appropriate to the requirements of the measurement being performed. For example, a scalar is sufficient for singles counting, while more sophisticated electronics are required for coincidence measurements. The quality of assay results is partially dependent upon the capabilities of equipment. The user will choose a suitable trade-off between detection efficiency, background shielding capabilities, equipment complexity, and equipment portability (weight, size, and number of pieces).

7.2 *Neutron Measurement Systems*—A quantitative holdup measurement may be performed using instrumentation that offers portability and simplicity of operation. The instrumentation typically includes a detector package with several ^{3}He detectors imbedded in cadmium-shielded polyethylene and supporting electronics in a portable package. The design of the neutron measurement system is dependent on the data quality objectives. In general, the size and weight of detector packages both increase with increased requirements for detection efficiency and background shielding.

7.2.1 Neutron measurement systems usually consist of one or more detector tubes (typically ^{3}He proportional counters) embedded in a moderating material (typically high-density polyethylene). The outer surfaces of the moderating material are typically covered with cadmium to stop thermal (highly moderated) room-scattered or environmental neutrons from entering the detector package. The measurement system is normally made somewhat directional by surrounding the sides and back of the detector package with a shield that consists of moderating material (typically high-density polyethylene).

7.3 *Detector Shielding:*

7.3.1 Design of a shield generally involves arriving at a compromise among several factors. Among these are a manageable weight versus detection efficiency and adequate shielding against background neutrons. Because of scattering, collimation of neutrons is generally not considered practical, and the detector packages have a somewhat directional but very wide field of view.

7.3.2 Detector shields can affect the efficiency of the detector package by scattering fast neutrons into the detector package, and each detector-shield assembly should be individually calibrated.

7.4 Detector positioning apparatus such as measuring and pointing devices or support stands to help attain reproducible geometry are recommended. Detector shield assemblies are typically too heavy to be moved and positioned without the aid of a positioning apparatus of some kind.

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 shall be followed.

8.1.2 Neutron detectors may use power-supply voltages typically as high as 2 kV for ^{3}He proportional counters. The power supply should be off before connecting or disconnecting high-voltage cables. Care should be taken to avoid damage to cables when moving the measurement systems through the measured facility.

8.1.3 Materials are used, for example, cadmium, that 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 with heavy instrumentation positioned in relatively inaccessible locations, as well as in elevated locations. Appropriate industrial safety precautions shall be taken to ensure personnel are not injured by falling objects, including the detector shield assembly, or that personnel do not fall while trying to reach the desired location.

8.2 *Technical Hazards:*

8.2.1 High-energy gamma rays can cause counts in some circumstances. Manufacturer instructions on the setup of electronics shall be followed rigorously to reduce this effect.

8.2.2 Electronic instability can impact assay results. For example, noise or microphonics can artificially increase measured count rates.

8.2.3 *Presence of Moderators*—If the holdup includes moderators (for example, oil or water) and appropriate allowances are not made, results will be adversely affected.

8.2.4 *Leakage Multiplication*—The fraction of source neutrons that escape from the item can be significantly different from unity. Careful modeling may be required to estimate these effects accurately.

8.2.5 *Background*—Lack of understanding of background effects on the measurement or incorrect background measurements may impact the results.

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

8.2.5.2 Neutrons can travel a long distance (for example, from a nuclear material storage location nearly 800 m away from the measured item). Because of scattering, simply pointing the detector away from the background source may provide unexpected results. An example is a measurement in which background taken with the detector pointed upwards toward open sky was 25 % higher than the measurement of the item.

8.2.5.3 Neutrons from adjacent items can scatter off of the measured item and into the detector.

8.2.5.4 Neutrons from an item behind the measured item can be scattered away from the detector making the item measurement lower than the background measurement.

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 a detailed measurement plan,

9.1.2 Preparation for measurements,

9.1.3 Perform the measurements,

9.1.4 Calculations and modeling (for example, specific neutron yield, detection efficiency, mass, uncertainty),

9.1.5 Estimation of measurement uncertainty (typically precision and bias), and,

9.1.6 Recording of data and results (**2-6**).

9.2 *Measurement Strategy/Plan Development:*

9.2.1 *Measurement Program Requirements*—Before the evaluation of an assay situation, specific information shall 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 the item or piece of equipment to be measured;

9.2.1.2 Radionuclide(s) 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; and

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, and

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

9.2.3 *Personnel and Procedures*—Note that 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 shall have adequate training, education, and experience. The definitions of adequate training, education, and experience can be found in Guide C1490. Development of measurement plans, strategy, and work instructions and the initial measurements generally require much more expertise than routine or subsequent remeasurements, 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 shall be performed before initiating measurements.

9.2.5 *Facility Evaluation*—The objective of the evaluation is to develop a measurement plan. This consists of several activities that are difficult to perform sequentially. Some are performed in parallel and iteration often is helpful. Each assay situation is unique. Information shall 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) or equipment, or both, to be assayed to gain an overview of the task at hand. Consider measurement geometry, other sources of radiation, moderating 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 firsthand information on current and historical process information and other important insights for consideration. Also, process operators and management that have participated in previous cleanout 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 used 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 and chemical composition remain constant throughout the areas to be assayed. This will include the radionuclides of interest as well as interfering radionuclides.

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.

9.2.5.7 Locations of holdup exceeding a predetermined activity level can be noted for later quantitative measurements.

9.2.5.8 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 *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 model can provide acceptable results for most items using the least amount of resources. However, nearly all facilities will also have special cases that require specialized models.

9.3.1 Several measurement techniques may be used. Each technique has advantages and disadvantages that shall 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 that optimizes the ability to determine the holdup quantities given the constraints on the effort (**4**, **5**).

9.3.2 Selection of assay calibration models includes assessment of factors such as 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 that 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 moderation problems may require use of contact or near-field measurements. A simple, item-specific model may allow results to be reached rapidly with minimal analysis and acceptable accuracy.

9.3.5 *Selection of Measurement Techniques*—Other factors that are generally determined for neutron measurements are leakage multiplication correction, distance between the source and the detector, and distance between contiguous measurements.

9.3.6 *Assay Plan*—The assay plan should provide clear instructions regarding everything affecting the equality of the holdup measurements. These considerations include support equipment, instrument settings, calibration and calibration checks, measurement locations, measurement distances, shielding, measurement times, background measurement, and measurement control (Guide C1009).

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

9.4 *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*—Before use, the apparatus shall 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 shall 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 shall 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.

9.4.3.2 If the measurement method and model use the item-specific approach, a standard or standard set that closely matches the actual holdup distribution will be required. Additionally, the standards will need to match the item leakage multiplication.

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 gamma-ray measurement techniques or 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 used. 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 C1009).

9.4.5.1 Three measurement control concepts can be used: check-source, measurements with no items present, and working source measurements. 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.

(1) Check-source Measurements—These measurements assure that the calibration of the measurement system has not changed. Sources are centered at a fixed distance from the detector face and measured for a fixed time. A check-source data set is established immediately following instrument calibration.

(a) 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 as a result of temperature, humidity fluctuations, or other reasons, additional measurements should be made.

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

(*3*) *Working Sources*—These sources, often a contaminated process equipment item, may be used to verify that instrument response has remained stable with time and verify adherence to procedures, proper operation of measurement instrumentation, and consistency of other parts of the measurement program. They also are helpful for evaluating the uncertainty caused by positioning of the equipment by the 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(s) of interest. 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 Background is typically a large source of uncertainty for neutron holdup measurements. Uncertainties arising from background subtraction can be significant because it is often difficult to accurately assess the proper background. Uncertainties in the background can be much larger than those arising from counting statistics. Significant background sources can include neighboring equipment, materials stored in the vicinity of the measurements, and in some cases, (for example, UF6 cylinder storage yards) materials stored as much as 800 m away.

9.5.2.1 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.

9.5.2.2 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. The response (count rate) is then influenced by the angular dependence of the instrument and by distance. Methods used include, but are not limited to, turning the detector in the opposite direction of the measurement, blocking the front face, and significantly increasing the distance between the detector and the measured item.

9.5.2.3 Plugs made of highly moderating materials that fit snugly against the detector face 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. When this technique is used, corrections for the background that is stopped by the plug and the source signal that is not stopped by the plug are normally required.

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 cleanout.

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

9.6.1 *Efficiency*—Calculations are performed as appropriate to the chosen calibration model and measurement techniques. An illustrative example is the MCNP modeling method. This method may be used to model each type of process equipment at a given source-to-detector distance. Provided the leakage multiplication is consistent from item to item, a single model can be used for each measurement of that type of process equipment.

9.6.2 *Specific Neutron Yield*—Isotopic distribution, chemical composition, and other information are used to estimate the number of neutrons produced per unit mass per unit time.

9.6.3 *Mass*—Efficiency, specific neutron yield, and net count rate(s) are used to estimate the mass of the nuclide or nuclides of interest, typically using a form of the following equation:

$$m = \frac{CR}{\varepsilon \cdot Y_n \cdot M_L}$$

where:

m = nuclide mass,
CR = net count rate,
ε = detection efficiency,
Y_n = specific neutron yield, and
M_L = Leakage Multiplication.

In some cases, multiple measurements of the same item are made from different measurement positions. When this is done, estimates of detection efficiency and nuclide mass are typically made for each measurement position. The estimates of nuclide mass are then evaluated to provide a best estimate for the item. The evaluation typically includes comparing the results for consistency with one another and with the modeled nuclide distribution. If the modeled nuclide distribution does not appear to be correct (for example, distribution was modeled as homogeneous, but the results for different measurement positions are significantly different from one another), the model may be modified and detection efficiencies re-computed. The best estimate for mass is usually either an average or uncertainty-weighted average of estimates from all measurement positions.

9.7 *Estimate Precision and Bias*—Because of 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 guide. 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.

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 leakage multiplication of the measured signal can cause a bias.

10.1.2 Uncertainties resulting from the use of overly simple models can cause a bias. By analyzing the data with a range of plausible assumptions, a bound can usually be placed on this effect.

10.1.3 Uncertainties in evaluating the background have caused large biases. The measurements performed often allow the uncertainty in the background to be assessed on a case-by-case basis.

10.1.4 Counting statistics associated with the item measurement generally impact the precision of the result and can be most easily addressed. Counting longer, aggregating results, and uncertainty propagation tools can be used to control and quantify these effects.

10.1.5 Of these four causes, counting statistics is easily controlled for all but the smallest holdup. 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 high.

10.1.6 Uncertainty contributions common to most in-situ measurements are listed in Table 1.

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

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 without changing measurement geometries can provide data for estimating precision.

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

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

10.4 *Bias*—It is not possible to specify succinctly the bias of the techniques described in this guide since each assay location or situation, with few exceptions, is unique. Biases as high as several hundred percent have been reported (**3-6**). One study

TABLE 1 Typical Contribution to Uncertainty in Neutron Holdup Measurements

Isotopic Composition or Enrichment	The isotopic composition of the measured deposit or material is influenced by process and facility history and may be a composite of different compositions. For a stable constant process, the desired isotopic composition may be determined from facility records, stream averages, or destructive analysis values. The isotopic composition may be accurately measured nondestructively with HPGe detector systems and commercially available gamma-ray isotopic analysis software. If the process has used a variety of enrichments or isotopic compositions, the uncertainty in the isotopic value will be increased. This uncertainty component can usually be readily determined or bounded and is often very small.
Deposit Chemical Composition	The chemical composition of the deposit affects (α, n) production and specific neutron activity for singles counting. The chemical composition may be determined or inferred from documented process history or from sampling and chemical analysis.
Deposit Geometry and Measurement Distance	The geometry of the deposit and source-to-detector distance affect detection efficiency. Modeling is typically used to estimate detection efficiency. The uncertainty of the location(s) or distribution of the deposit within the measured equipment also contribute to this uncertainty, which may be bounded using equipment drawings and further refined by process knowledge of where deposits typically form for a given type of equipment. Modeling several potential geometries and deposit distributions can provide information needed to estimate potential differences in detection efficiency and the magnitude of this uncertainty.
Calibration Uncertainty	This is a systematic uncertainty for all of the measurements with a single calibration. If calibrations are repeated often during a measurement campaign this uncertainty in some cases becomes a random uncertainty.
Leakage Multiplication	For all but the largest uranium deposits, leakage multiplication is typically very close to one and does not add significantly to the total measurement uncertainty. For plutonium deposits, multiplication can be a significant source of uncertainty.
Background	Background is typically a large source of uncertainty for neutron holdup measurements. Uncertainties arising from background subtraction can be significant because it is often difficult to accurately assess the proper background. Uncertainties in the background can be much larger than those arising from counting statistics. Significant background sources can include neighboring equipment, materials stored in the vicinity of the measurements, and in some cases (for example, UF_6 cylinder storage yards) materials stored as much as 800 m away.
Cosmic-Ray Spallation	Some process equipment can be extremely large and heavy, especially in uranium processing facilities. This can cause a significant background from cosmic-ray spallation. Annex A2 provides a method for estimating this effect.
Counting Statistics	Counting statistics can be a significant contributor to total measurement uncertainty, as specific neutron activity is low, background is relatively high, and the signal-to-noise ratio is often only slightly greater than one. Relatively long counting times are often needed to reduce counting statistics to meet data quality objectives for minimum detectable activity.

showed no correlation between gamma-ray and neutron measurements of the same items, with overall uncertainty of greater than 100 % (**3**). 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 non-uniformity of the deposit, errors in estimation of corrections, incorrect modeling, incorrect background subtraction, plus incorrect assumptions regarding geometry, isotopic composition, chemical form, and 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. An NDA professional's advice should be sought to determine the correct approach for the measurement situation.

10.4.2 After adjusting the calculational models based on the cleanout values, overall uncertainty as small as 5 % has been reported (**4**).

10.4.3 Experience indicates 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 high compared to the actual value.

11. Keywords

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

ANNEXES

(Mandatory Information)

A1. SPECIFIC NEUTRON YIELD

INTRODUCTION

Specific neutron yield, or number of neutrons generated per unit time per unit mass of the nuclide(s) of interest, is an important parameter for passive neutron holdup measurements. For both uranium and plutonium, it is affected by chemical composition. This annex presents values suggested for use in estimating specific neutron yield.

A1.1 Uranium

A1.1.1 In uranium enrichment facilities, the assumption is often made that holdup material is present in the form of UO_2F_2. The specific (α, n) contribution due to F has historically been subject to a large uncertainty witnessed by the spread in reported values of notionally higher quality. Recent work by LaFleur et al (**7**) appears to have settled this issue with a measured value for ^{234}U, the largest contributor to (α, n) contribution in enriched uranium, with an uncertainty of about 2 % at 1σ. The shape of the thick target yield curve may be used along with the decay data of the other U-nuclides to estimate the specific yields for them (**8**). Uncertainties in the alpha decay half-life and alpha branching ratios introduce only a small relative uncertainty, typically on the order of a fraction of a percent.

A1.1.2 The specific (α, n) contribution due to O is relatively small and is also well known (**8**). For pure stoichiometric UO_2F_2, the uncertainty in the calculated oxygen contribution is estimated to be on the order of 3 %.

A1.1.3 The spontaneous fission (SF) contribution can be estimated from measured SF half-lives (**9**) and combined with the evaluated mean number of prompt neutrons emitted per fission (**10**) and delayed neutron estimates (**11**). For ^{238}U, which is usually the dominant source of SF emissions, the specific SF yield estimated in this way is currently known to be about 1.7 % at 1σ. For ^{234}U and ^{235}U where the data is not so extensive and systematic relationships are needed to fill in the gaps, the uncertainties are higher at about 10 % and 30 %, respectively.

A1.1.4 From this basis, Table A1.1 was constructed.

A1.1.5 Impure or moist, or both, UO_2F_2 may have a markedly different (α, n) yield and the advice of a subject matter expert should be sought. For example, the (α, n) yield for $UO_2F_2{\cdot}2H_2O$ is approximately 20 % less than for anhydrous UO_2F_2 (**12**).

A1.1.6 Scaling factors can be used to estimate specific neutron yield for compounds other than UO_2F_2. The recommended scaling factor for UF_6 is 2.332 (that is, UF_6 (α, n) yield is about 2.332 times larger than for UO_2F_2) (**8**).

A1.2 Plutonium

A1.2.1 Recommended specific neutron activities for PuF_4 are provided in Table A1.2. Uncertainties in PuF_4 estimates are: ^{238}Pu 0.102 %, ^{239}Pu 0.124 %, ^{240}Pu 0.106 %, ^{241}Pu 2.164 %, ^{242}Pu 0.532 %, and ^{241}Am 0.116 %. This is due to uncertainty in half-lives and branching ratios and does not include uncertainty in (α, n) yield.

TABLE A1.1 Specific Neutron Yields for Uranium Isotopes

Nuclide	F in UO_2F_2 n/s/g of nuclide (RSD ±2 %	O in UO_2F_2 n/s/g of nuclide (RSD ±3 %)	Spontaneous Fission n/s/g of nuclide
^{234}U	1.971×10^{2}	2.261×10^{0}	6.71×10^{-3} ± 10 %
^{235}U	3.820×10^{-2}	5.272×10^{-4}	1.05×10^{-5} ± 30 %
^{238}U	3.934×10^{-3}	6.047×10^{-5}	1.334×10^{-2} ± 1.7 %

TABLE A1.2 Specific Neutron Yields for Plutonium Isotopes

Nuclide	PuF_4 (8) n/s/g of nuclide	Spontaneous Fission (13) n/s/g of nuclide
^{238}Pu	3.094×10^{6}	2.65×10^{3}
^{239}Pu	7.798×10^{3}	1.48×10^{-2}
^{240}Pu	2.880×10^{4}	1.04×10^{3}
^{241}Pu	2.309×10^{2}	1.72×10^{-3}
^{242}Pu	3.650×10^{2}	1.72×10^{3}
^{241}Am	6.150×10^{5}	1.63×10^{0}

A2. COSMIC-RAY SPALLATION PRODUCTION SCALING

A2.1 The specific neutron production rate, α in units of n/s/g, resulting from cosmic rays has been found to scale with molar mass, A, g/mol, approximately in the following way:

$$\alpha \approx kA^{b}$$

where k is a constant of proportionality, determined at the location (altitude, overburden, and prevailing weather conditions) often with a surrogate such as Pb, and $b \approx (0.55 \pm 0.13)$ (14).

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