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Standard Guide for Nondestructive Assay Measurements[1]

This standard is issued under the fixed designation C 1592; 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 is a compendium of Good Practices for performing measurements of radioactive material using nondestructive assay (NDA) instruments. The primary purpose of the guide is to assist its users in arriving at quality NDA results, that is, results that satisfy the end user's needs. This is accomplished by providing an acceptable and uniform basis for the collection, analysis, comparison, and application of data. The recommendations are not compulsory or pre requisites to achieving quality NDA measurements, but are considered contributory in most areas.

1.2 This guide applies to the use of NDA instrumentation for the measurement of nuclear materials by the observation of spontaneous or stimulated nuclear radiations, including photons, neutrons, or the flow of heat. Recommended calibration, operating, and assurance methods represent guiding principles based on current NDA technology. The diversity of industry-wide nuclear materials measurement applications and instrumentation precludes discussion of specific measurement situations. As a result, compliance with practices recommended in this guide must be based on a thorough understanding of contributing variables and performance requirements of the specific measurement application.

1.3 Selection of the best instrument for a given measurement application and advice on the use of this instrument must be provided by a qualified NDA professional following guidance provided in Guide C 1490. This guide is to be used as a reference, and to supplement the critical thinking, professional skill, expert judgement, and experimental test and verification needed to ensure that the instrumentation and methods have been properly implemented.

1.4 The intended audience for this guide includes but is not limited to Management, Auditor Support, NDA Qualified Instrument Operators, NDA Technical Specialists, and NDA Professionals.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

C 859 Terminology Relating to Nuclear Materials

C 1030 Test Method for Determination of Plutonium Isotopic Composition by Gamma-Ray Spectrometry

C 1133 Test Method for Nondestructive Assay of Special Nuclear Material in Low-Density Scrap and Waste by Segmented Passive Gamma-Ray Scanning

C 1207 Test Method for Nondestructive Assay of Plutonium in Scrap and Waste by Passive Neutron Coincidence Counting

C 1215 Guide for Preparing and Interpreting Precision and Bias Statements in Test Method Standards used in the Nuclear Industry

C 1221 Test Method for Nondestructive Analysis of Special Nuclear Materials in homogeneous Solutions by Gamma-Ray Spectrometry

C 1254 Test Method for Determination of Uranium in Mineral Acids by X-ray Fluorescence

C 1268 Test Method for Quantitative Determination of Americium 241 in Plutonium by Gamma-Ray Spectrometry

C 1316 Test Method for Nondestructive Assay of Nuclear Material in Scrap and Waste by Passive-Active Neutron Counting Using a ^{252}Cf Shuffler

C 1455 Guide for Nondestructive Assay of Special Nuclear Material Holdup Using Gamma-Ray Spectroscopic Methods

C 1458 Test Method for Nondestructive Assay of Plutonium, Tritium and ^{241}Am by Calorimetric Assay

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

C 1493 Test Method for Non Destructive Assay of Nuclear Material in Waste by Passive and Active Neutron Counting Using a Differential Die Away System

[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.

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[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.

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C 1514 Test Method for Measurement of ^{235}U Fraction Using the Enrichment Meter Principle

3. Terminology

3.1 Definitions presented here are confined to those terms not defined in common nuclear materials glossaries/references or whose use is specific to this application. The use of statistical terms is consistent with the definitions in American National Standard Statistical Terminology and Notation for Nuclear Materials Management, N15.5-1972. Some of those definitions are repeated here for convenience to the reader.

3.2 *Definitions:*

3.2.1 *(α n) reactions*—(α, *n*) reactions occur when energetic alpha particles collide with low atomic number nuclei, such as O, F, or Mg, producing single neutrons.

3.2.2 ^{240}Pu *effective mass*—m_{eff} is the mass of ^{240}Pu that would produce the same coincident, or total, neutron response in the instrument as the assay item, all other factors remaining unchanged. It is correlated to the quantity of even mass isotopes of plutonium in the assay item.

3.2.3 *absorber foils*—thin metal foils that are used to reduce the contribution of low-energy gamma rays to the overall count rate.

3.2.4 *accidentals*—the accidental or random summing of neutrons generate a signature like that from true or Real coincidences. For shift register pulse train deconvolution the number of neutrons detected in the (*A*) gate period following the initial detection of each neutron during the selected count time *t*. This is a measured quantity.

3.2.5 *accuracy—(1)* bias; *(2)* the closeness of a measured value to the true value; and *(3)* the closeness of a measured value to an accepted reference or standard value.

3.2.6 *assay*—to determine quantitatively the amount of one or more nuclides of interest contained in an item, or the result of such a determination.

3.2.7 *background*—extraneous signal superimposed on the signal of interest.

3.2.8 *benign matrix*—bulk material that has no effect on the result of the measured parameter.

3.2.9 *bias*—a constant positive or negative deviation of the method average from the correct value or accepted reference value.

3.2.10 *calibration*—the determination of the values of the significant parameters by comparison with values indicated by a reference instrument, by a set of reference standards or modeled parameters. **C 859**

3.2.11 *certification*—a written declaration from a certifying body or its legitimate designee that a particular measurement process complies with stated criteria, or a measured item has the stated characteristics.

3.2.12 *coincident neutrons*—two or more neutrons emitted simultaneously from single event, such as from a nucleus during fission.

3.2.13 *collimator*—usually constructed of lead or tungsten, a collimator serves to define a gamma-ray detector's horizontal and vertical viewing angles and to shield the detector from ambient radiation.

3.2.14 *confidence interval*—the range of values, calculated from an estimate of the mean and standard deviation, which is expected to include the population mean with a stated level of confidence.

3.2.15 *control chart*—a graphical plot of test results with respect to time or sequence of measurement together with limits in which they are expected to lie when the system is in a state of statistical control.

3.2.16 *control limits*—the limits shown on a control chart beyond which it is highly improbable that a point could lie while the system remains in a state of statistical control.

3.2.17 *corrections*—techniques that are part of the data analysis or method, which compensate for the effects of variables that interfere with the measurement and degrade accuracy. These corrections account for such things as matrix material, lumps, heterogeneity, dead time, and background.

3.2.18 *dead time*—the period following the detection of an event during which the system cannot register a subsequent event. Dead time is usually expressed as a percentage of elapsed time.

3.2.19 *differential die away technique (DDT)*—an NDA technique for characterizing the prompt neutrons from fissionable isotopes in scrap and waste using a neutron generator interrogation source.

3.2.20 *good measurement practice*—an acceptable way to perform some operation associated with a specific measurement technique that is known or believed to influence the quality of a measurement (a way to perform some operation associated with a specific NDA technique in a manner that meets the quality requirements of a measurement).

3.2.21 *holdup*—the amount of nuclear material remaining in process equipment and facilities after the in process material, stored materials and product are removed.

3.2.22 *homogeneous matrix*—the degree to which the matrix materials are spread uniformly throughout the item container. Non homogeneous matrices are referred to as heterogeneous.

3.2.23 *in process material*—the nuclear material in a process stream, excluding holdup.

3.2.24 *item*—nuclear material in a container or other suitable configuration for assay.

3.2.25 *lower limit of detectability*—a stated limiting value which designates the lowest concentration, mass, or activity that can be detected with confidence and which is specific to a particular measurement. **C 859, C 1215**

3.2.26 *low level waste*—waste that is not defined as transuranic or high level waste. **DOE order 435.1**

3.2.27 *matrix*—the material, which comprises the bulk of the item, except for the special nuclear material and the container. This is the material in which the special nuclear material is embedded.

3.2.28 *matrix-specific calibration*—uses a calibration matrix similar to the matrix to be measured. No matrix correction factors are used. This calibration is generally not appropriate for other matrices.

3.2.29 *modeling*—the use of mathematical techniques to simulate a measurement process or alternatively the process of creating a physical mock up of a measurement.

3.2.30 *neutron absorbers*—materials, which have relatively large thermal-neutron absorption cross sections. Absorbers with the largest cross sections are commonly known as neutron poisons. Some examples are lithium, boron, cadmium, and gadolinium.

3.2.31 *neutron moderators*—materials which slow down neutrons through elastic scattering or inelastic interactions. Materials containing large amounts of low atomic weight materials, for example, hydrogen are highly moderating.

3.2.32 *neutron multiplication*—multiplication takes place when a neutron interaction yields more than one neutron as a product. Induced fission is the primary mechanism for neutron multiplication, however (*n*, 2*n*) interactions are also multiplication events.

3.2.33 *nondestructive assay (NDA)*—the observation of spontaneous or stimulated nuclear radiations, interpreted to estimate the content of one or more nuclides of interest in the item assayed, without affecting the physical or chemical form of the material.

3.2.33.1 *active assay*—assay based on the observation of radiation(s) induced by irradiation from an external source.

3.2.33.2 *passive assay*—assay based on the observation of naturally occurring or spontaneous nuclear radiation(s).

3.2.34 *nuclide*—an atomic species characterized by the composition of its nucleus, that is, by the number of protons and neutrons it contains.

3.2.35 *passive neutron coincidence counting*—a technique used to measure the rate of coincident neutron emission in the assay item. The terminology used in this test method refers specifically to shift-register electronics.

3.2.36 *Poisson assumption*—for counting measurements, it is assumed that the net counts in a fixed period of time follow a Poisson distribution. This assumption can be verified by comparing the observed standard deviation of a series of measurements on an item with the square root of the average number of counts. If the Poisson assumption is correct, these numbers should be equal within random error.

3.2.37 *precision*—a generic concept used to describe the dispersion of a set of measured values. Measures frequently used to describe precision are standard deviation, relative standard deviation, variance, repeatability, reproducibility, confidence interval, and range. (See Guide C 1215 for a more complete discussion of precision.)

3.2.38 *procedure*—a set of systematic instruction for using a method of measurement or of the steps associated with the method.

3.2.39 *qualitative analysis*—an analysis in which some or all of the components of an item are determined. A measurement in which the amount of one or more components of an item are determined.

3.2.40 *radioactive emissions*—alpha, beta, gamma-ray, x-ray, heat, and neutron emissions from spontaneous fission, induced fission, or delayed neutron emission following beta decay.

3.2.41 *radioactive scrap*—materials that contain sufficient quantities of source or special nuclear material to be worthy of recovery. **C 859**

3.2.42 *radioactive waste*—items containing radioactive materials not currently considered useful or economically recoverable. **C 859**

3.2.43 *random error*—the chance variation encountered in all measurement work, characterized by the random occurrence of deviations from the mean value. **C 1215**

3.2.44 *rate loss correction*—a correction for count rate related losses that are used for some gamma-ray NDA techniques. The correction may use radioactive sources with gamma-ray energies lower than the gamma-ray from the nuclide of interest or a pulser.

3.2.45 *reals*—this quantity is the difference between the (*R*+*A*) and (*A*) quantities.

3.2.46 *reals plus accidentals*—the number of events detected in the (*R*+*A*) gate period following the initial detection of each neutron associated with neutron counting. This is a measured quantity during the count time.

3.2.47 *repeatability*—the within group dispersion of several groups of measurements. **C 1215**

3.2.48 *replicate*—a counterpart of another measurement. It is the general case for which duplicate, consisting of two measurements, is the special case.

3.2.49 *reproducibility*—the between group dispersion of several groups of measurements. **C 1215**

3.2.50 *sample*—a portion of a population or lot. In the context of NDA measurements, it may consist of measurements of items that are part of a larger group that could have been considered.

3.2.51 *secular equilibrium*—the state of equilibrium that exists when series of radioisotopes have equal and constant activity levels. Secular equilibrium is established when the half life of the parent is much greater than that of the decay products.

3.2.52 *segmented gamma scanner*—a nondestructive assay technique used to measure the gamma-ray emissions from low-density scrap and waste packaged in cylindrical containers. The technique involves independent measurements of the vertical segments of the container and may incorporate corrections for count rate losses and matrix attenuation.

3.2.53 *self-attenuation*—the attenuation of emitted radiation by the emitting material itself.

3.2.54 *sensitivity*—the capability of methodology or instrumentation to discriminate between items having differing concentrations or containing differing amounts of a radioactive material.

3.2.55 *shift-register-based coincidence circuit*—an electronic circuit for determining totals *T*, Reals plus Accidentals (*R*+*A*), and accidentals (*A*) in a selected count time (*t*) during neutron counting.

3.2.56 *shuffler*—an NDA technique for characterizing the delayed neutrons from fissionable isotopes in scrap and waste using a ^{252}Cf interrogation source.

3.2.57 *special nuclear material (SNM)*—Plutonium, ^{233}U, uranium enriched in ^{233}U or ^{235}U to greater than its natural abundance, and any other materials defined as SNM under the Atomic Energy Act of 1954, as amended. This term does not include source materials. **C 859**

3.2.58 *standard:*

3.2.58.1 *calibration standard*—an item sometimes physically and chemically similar to the items to be assayed, for which the mass of the nuclide(s) of interest and all properties to which the measurement technique is sensitive are known.

3.2.58.2 *working standard*—an item used to check the performance of an NDA instrument, nominally representative of the items to be assayed, and fabricated and handled to ensure its internal integrity so that deviations in its measured response can be attributed to the instrument.)

3.2.59 *total measurement uncertainty (TMU)*—an estimated parameter, either mass, activity, concentration, or fractional, used to quantify the overall confidence in the assay result at a prescribed level including all sources of precision and bias. The TMU is qualified by the assumptions of the error propagation model.

3.2.60 *totals*—the total number of neutrons detected during the count time. This is a measured quantity.

3.2.61 *traceability*—the property determined by a measurement which can be related to appropriate standards, generally national or international standards, through an unbroken chain of comparisons.

3.2.62 *transmission source*—a radioactive source external to the item being measured that is used to determine the attenuation of gamma rays of interest by the matrix material in the item.

3.2.63 *transuranic waste (TRU waste)*—as defined in DOE Order 5820.2 and DOE Order 435.1, transuranic waste is radioactive waste containing alpha-emitting isotopes with atomic number greater than 92 and half-life greater than 20 years, and with activity concentrations greater than 100 nCi per gram of waste at the time of the measurement.

3.2.64 *uncertainty*—a generic term describing the inability of a measurement process to determine the correct value. (Alternate definition: Parameters associated with the result of a measurement that characterizes the dispersion of values that could reasonable be attributed to the measurement.)

3.2.65 *validation*—an evaluation that shows the quality assurance and quality control mechanisms are in place and functioning properly to ensure that the waste characterization information is collected and analyzed in a manner described by procedures and meets assigned data quality objectives.

3.2.66 *verification*—an evaluation of the critical item characteristics to ensure the collected characterization data represents the true characteristics of the sample population to an acceptable degree of accuracy and precision.

3.2.67 *waste acceptance criteria (WAC)*—the set of requirements pertaining to a waste item that must be satisfied before it can be shipped to a designated facility or disposal.

4. Significance and Use

4.1 Good NDA measurement practices are described in this guide. The application of the material provided in this guide should be determined on a case by case basis. Not all elements are required for all applications.

4.2 Nondestructive assay measurements are typically performed when the items measured or goals of the measurement program favor NDA over destructive analysis. NDA is typically favored when collecting a representative sample of the item is difficult or impractical (for example, scrap and waste items), personnel exposure would be significant, spread of contamination from sampling would occur, generation of secondary waste must be minimized, the weight and/or tare weight of the item cannot easily be determined (for example, in place process equipment), rapid turn around of the measurement results is needed, or the NDA measurement is significantly less expensive than the equivalent destructive analysis.

4.3 The principles provided in this guide should be used to determine which type of measurement is best suited to the measurement application. This determination involves consideration of the characteristics of the items to be measured, as well as the goals of the measurement program.

4.4 This guide applies to the suite of NDA instruments and measurement methods, many of which are described in detail in Refs **(1)** and **(2)**.[3] A partial listing of measurement methods and applicable use references is provided in 5.2. It is incumbent upon the user to seek additional guidance within ASTM method-specific standards, as this guide does not take precedence. Additional information on specific methods is best found in technical meeting transactions, journals, commercial application notes, and NRC/DOE publications.

4.5 This guide may be applied to many situations spanning the range of nuclear materials from product through waste. Typical applications include: the measurement and characterization of transuranic wastes, low-level wastes, and mixed wastes; the determination of radioactivity below some regulatory threshold; estimated for non detected radionuclides, the measurement of safeguarded nuclear materials; shipper receiver confirmation; confirmation of nuclear material inventory; support of nuclear criticality safety evaluations; measurement of holdup of special nuclear material in process systems; support of decontamination and decommissioning activities; and in-situ analyses of facilities, glove-boxes, hot cells, and the environment prior to and following demolition.

4.6 When applied to measurement of waste, this guide should be used in conjunction with a waste management plan that segregates the contents of assay items into material categories according to some or all of the following criteria: bulk density of the waste, chemical forms of the radioactive constituents and matrix, (α, n) neutron intensity, hydrogen (moderator) and absorber content, thickness of fissile mass(es), and the assay item container size and composition. Each matrix may require a different set of calibration standards and may have different mass calibration limits. The effect on the quality of the assay (that is, minimizing precision and bias) can significantly depend on the degree of adherence to this waste management plan.

4.7 This guide addresses elements of good practice such as; nuclear measurement instrumentation and its care; common hazards; facility readiness and requirements to support the NDA equipment; project scoping, requirements and objectives; assembly and deployment of the instrument; calibration and test; computational modeling to augment physical testing; measurement validation; preventive maintenance; and the measurement control program.

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

5. Good Practice

5.1 *Introduction*—NDA measurements of nuclear material are performed to determine the relative or absolute abundance of one or more nuclides. Typically, such a determination is made by comparing the observed response of an unknown amount of material to the response of one or more known standards by means of a functional relationship established by calibration. NDA refers to the qualification and quantification of radionuclides using instrumentation capable of detecting a feature of the radioactive-decay process. These features include such radioactive emissions as alpha, beta, gamma-ray, x-ray, heat, and neutron emissions from spontaneous fission, induced fission, or delayed neutron emission following beta decay.

5.2 The primary goal of NDA measurements is to arrive at a quality result, that is, one that satisfies the user's measurement needs. Adequately analyzing problems and applying appropriate measurement techniques support this goal.

5.3 Each NDA technique has advantages and limitations that must be judged against the specific requirements of the intended applications. No single technique can satisfy all requirements. It is the responsibility of the user to consider the potential problems, and select the proper balance of measurement capability and desired precision and accuracy for the specific application.

5.4 The observed response of an NDA system shows sensitivity to a wide variety of factors that can bias the assay result. By careful selection of the measurement technique, attention to potential sources of error, implementation of operational procedures to control item categorization and packaging, operator training and instrument maintenance, supplemental measurements and calculations, and proper organization and evaluation of test data, the quality of assay results can be optimized.

5.5 Because performance requirements for NDA systems are application dependent, only general guidance for the selection of a system can be provided. If more than one technique can satisfy the specific measurement requirements, other considerations such as economics, ease of operation, and availability of instrumentation will ordinarily determine the choice of a system. The following parameters are among those that should be considered when selecting NDA measurement systems:

(a) The radionuclides to be measured, including the expected range of assays and interferences that may arise between radionuclides,

(b) The physical form (particle size, particle density, radioactive material distribution, etc.),

(c) The matrix (for example, pure product, oily waste, dry waste, degree of heterogeneity, average density, etc.),

(d) The container and packing material (for example, size, wall thickness, mass, wall material),

(e) Environmental conditions,

(f) Measurement quality objectives,

(g) The degree to which parameters affecting measurement results are known,

(h) Location(s) at which measurements are needed,

(i) Costs (instrument, set up, and operating costs),

(j) Availability of instrumentation,

(k) System maintenance requirements (reliability, stability, ruggedness, etc.),

(l) Training requirements,

(m) Ease of operation,

(n) Program schedule, and

(o) Item throughput.

5.6 NDA methods are often nuclide sensitive rather than element sensitive. Frequently the reaction of interest is possible in more than one species of nucleus present. Determination of the elemental content of an item from a measurement of radiations emitted by isotope(s) of the elemental species and, in some cases, by their decay products requires knowledge of the relative radionuclide composition of the item assayed.

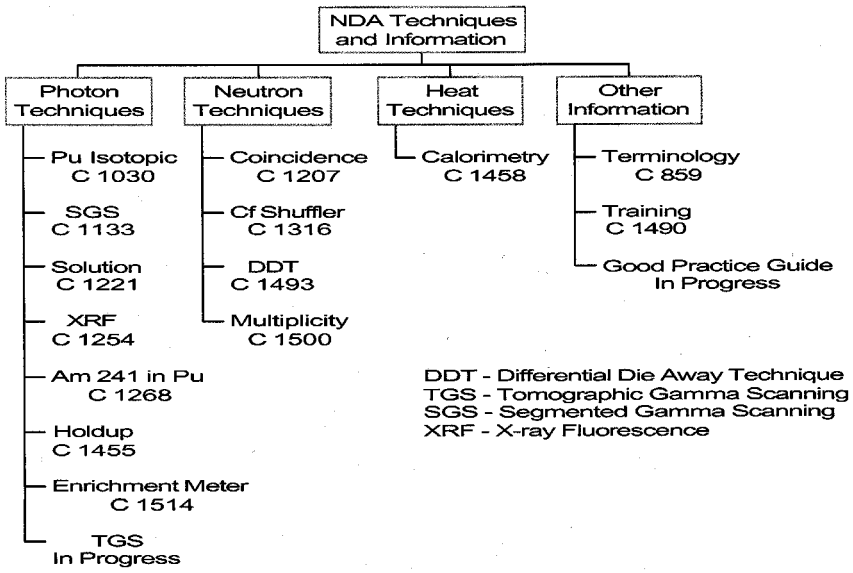

FIG. 1 NDA Techniques

5.6.1 Many of the approaches to specific NDA measurement techniques are described by ASTM Standards as shown in Fig. 1. A listing of applicable ASTM and ANSI standards is provided in Section 7, Test Methods. Other standards may also be considered.

5.6.2 *Neutron Measurement Techniques*—Neutron techniques are based on the detection of neutrons, which are emitted with various energies. Neutron energies are generally not measured. A passive neutron measurement is made when the neutrons measured are a result of spontaneous fission, self induced fission, or (α, *n*) reaction. Passive neutron assay systems are usually more effective for plutonium than for uranium, although applications for both exist. An active measurement is performed when the measured neutrons are the result of induced fission. The quantity of a particular isotope may be obtained by measuring unusually low or high emission rates, distinctive time distributions, or markedly different energy spectra. To establish the quantity of radionuclide of interest from the directly observable neutron assay result(s) relative isotopic information is necessary. Correction or allowances may be needed for:

(a) (α, *n*) contaminants,

(b) Hydrogen content,

(c) Neutron moderation and absorption (poisons),

(d) Container wall effects,

(e) Item size,

(f) Influence of uranium on plutonium assay,

(g) Source self-shielding,

(h) Non-uniformity in source/matrix distribution as it relates to neutron moderation and absorption,

(i) Unexpected neutron generating radionuclides,

(j) Chemical composition,

(k) System dead time,

(l) Item size (physical dimensions and amount of fissionable material),

(m) Measurement geometry,

(n) Background radiation,

(o) Density, and

(p) Neutron multiplication.

5.6.2.1 *Passive Neutron Counting*:

(1) Total neutron counting serves as a suitable technique if the material to be assayed is homogeneous with respect to all attributes affecting the measurement, if it contains little or well characterized (α, *n*) target material, and if the nuclidic ratios are well known. The primary strengths of total neutron counting are that it usually does not depend on the use of external sources of radiation and that passive neutrons are of sufficient energy to escape from most items without significant attenuation. The costs for total neutron emission measurement programs are often considerably less than for active measurement techniques. In addition, because external neutron sources are not required, risk of personnel exposure to radiation is generally lower for total neutron assay. The primary disadvantages of total neutron assay relative to active neutron assay are that counting rates are often lower and contaminants contribute to the totals count rate resulting in a bias. The presence of (α, *n*) target material can result in a bias unless the relative amount of this material and its yield are well known and appropriate compensation is included in quantity estimates.

(2) Passive Coincidence-neutron counting is a viable technique for the measurement of ^{240}Pu effective mass or ^{238}U in low enriched uranium. Isotopic ratios are necessary to compute the grams of element. Coincidence neutron counting is less sensitive to many of the biases typical of total neutron counting because their contribution (for example, the presence of [α, *n*] target material) is eliminated. In addition, spontaneous fission of ^{244}Cm interferes with the measurement of ^{240}Pu effective mass.

(3) Multiplicity counting is a viable assay technique for plutonium in cases where sufficient counting precision may be obtained for higher order coincidences. In principle, the technique does not require representative standards, but they are often used to provide corrections to assays. It provides improved accuracy over conventional coincidence counting in cases where the measured items are impure or heterogeneous and the multiplication and/or (α, *n*) yield are not known prior to the measurement. The precision is usually poorer because of lower count rates for the higher moments. It can be used to reduce cosmic ray background even when the count rates for the higher moments are low.

5.6.2.2 Active assay by neutron interrogation is applicable when ^{235}U is present or when passive signals are weak. Selection of an appropriate interrogating-neutron spectrum is important. Active techniques are sometimes used when the uncertainty in the passive result is unacceptable. Costs may be significantly higher than for passive assay systems. In addition, the matrix in which the measured nuclides are contained is often an important consideration.

5.6.2.3 Thermal neutrons can be used for active neutron assay systems if they can adequately penetrate the item. The presence of thermal-neutron absorbers such as gadolinium (Gd) in light-water-reactor (LWR) fuel may preclude the use of a thermal spectrum. Thermal-neutron interrogation may be possible for small items with high moderation, for example, hydrogen (H) content (for example, solutions). Interrogation with thermal neutrons offers the advantage of higher detection sensitivity because of increased fission cross sections at low neutron energies in fissile material.

5.6.2.4 For the assay of uranium-bearing items of high density, interrogation by neutrons having energies greater than thermal is recommended. Interrogating-neutron spectra can originate from various sources such as spontaneous fission isotopes, neutron generators or accelerators.

5.6.2.5 A major problem in active neutron assay is differentiation between the interrogating radiation and the stimulated response radiation. Ideally, the detector should be insensitive to the interrogating radiation. Although total insensitivity is seldom achieved, the amount of interrogating radiation detected can be reduced by several techniques. These techniques include using an energy-biased detector, coincidence counting, timing, and shielding.

5.6.3 *Calorimetric Assay*—Applications of calorimetry to NDA refer to the measurement of heat flow from radioactive decay. Calorimetric assay typically provides assays with very good precision and low bias. Typical assay times range from 4

to 24 hours. Typically calorimeter chambers are 8 in. diameter or less. To estimate the quantity of radionuclide of interest present, the effective specific power, or amount of heat generated per unit mass per unit time, must be determined from knowledge of the item's isotopic composition. For plutonium, this is typically accomplished using high-resolution gamma-ray spectrometry. Corrections or allowances may be needed for:

(a) Heat-generating contaminants,

(b) Isotopic composition,

(c) Chemical reactions which produce or consume heat,

(d) Phase changes which produce or consume heat,

(e) Weight of the measured item (dependent on calorimeter design),

(f) Endpoint detection and prediction methods are usually dependent on heat transfer characteristics of the item,

(g) Heat transfer characteristics of the item's packaging (dependent on end-point detection method), and

(h) Drift in Bridge Potential (or baseline power for servo-control method).

5.6.4 Photon techniques are based on the detection of gamma or x-rays that are emitted with discrete energies characteristic of specific isotopes. The intensity of photons of a specific energy is related to the quantity of a particular isotope. The relative intensity of gamma rays from different isotopes can be related to the relative abundance of those isotopes. There are a variety of detectors available, which generally span the range of efficiency and resolution from relatively high to low.

5.6.4.1 *Isotopic Composition*—Gamma ray spectrometry may be used to determine isotopic composition (Test Methods C 1030, C 1268). Isotopic composition from gamma ray spectrometry is often used to support both calorimetry (Test Method C 1458) and neutron techniques (Test Methods C 1207, C 1493, C 1500, and C 1316), as well as for other applications.

5.6.4.2 *Quantitative Assay*—Gamma ray spectrometry is used for quantitative assay of specific isotopes in situations where attenuation by the container wall, by the item's matrix, and self attenuation by the radionuclides is not excessive, or can be accurately estimated. Estimates of attenuation are typically obtained from process knowledge, item density, transmission, or differential peak analysis.

(1) Transmission Corrected Assay—In some cases, a transmission source is used to provide an estimate of the matrix attenuation. This typically results in a more accurate assay than uncorrected assays.

5.6.4.3 Correction or allowances may be needed for:

(a) Lumping (self absorption),

(b) Radial/axial non-uniformity of the radioactive source material in the item,

(c) Matrix heterogeneity,

(d) Non representative calibration standards,

(e) Attenuation,

(f) Low signal to noise ratio,

(g) Signal distortion,

(h) Dead time correction,

(i) Measurement geometry,

(j) Item size (physical dimensions),

(k) Container packaging and matrix attenuation,

(l) Background radiation,

(m) Interfering radiation, and

(n) Decay of radioactive sources used to routinely test the stability/functionality of a measurement system, transmission sources, and rate base correction sources.

5.7 Specific radioisotopes may not be directly quantifiable by certain NDA measurement techniques in given situations. However, when the abundance of an unobservable radionuclide of interest is known (either from independent analysis or established correlation functions) relative to that of one or more radionuclides that can be directly measured, it is possible to infer its quantity. Subject matter experts must address the validity and accuracy of the estimate.

5.8 *Calibration*—Calibration provides a mathematical relationship to correlate detector response with characteristics of the measured item. Methods used for calibration are specific to the NDA measurement technique. In general, calibrations are performed in such a manner that overall calibration uncertainty is substantially lower than the target uncertainty for item measurements. The amount of effort expended on calibration should be associated with the quality objectives of the measurement results for items of unknown content (for example, a 0.1 % calibration uncertainty is not necessary for a measurement system that will produce results with 50 % total uncertainty). The data quality objectives are often, in turn, driven by regulatory, economic and ease of operation considerations. Some considerations that apply to calibration methods include:

(a) Determining the intrinsic system response,

(b) Assessing correction methods (for example neutron moderation, attenuation, absorption, geometry, self attenuation),

(c) Measuring instrument repeatability,

(d) Determining conditions that bias results (and the magnitude of the biases), and

(e) Determining total calibration uncertainty.

5.8.1 Calibration Standards should be selected carefully. It is not always necessary for NDA calibration standards to bracket the anticipated system measurement range. The selected standards, however, should have characteristics that are the same as items to be measured with respect to parameters that affect the measurement results. Standards should be constructed so as to eliminate the possibility of a redistribution of the radionuclide content during use. Considerations for selection of calibration standards include:

(a) Standard type (element, state, etc.),

(b) Durability and stability under routine use,

(c) NDA measurement method,

(d) Container size,

(e) Matrix attenuation properties,

(f) Gamma self-attenuation and neutron self shielding properties,

(g) Emission rate for radiation of interest,

(h) Number of standards required,

(i) Replacement interval for standards with short half life, are subject to chemical instability, or pressure build up,

(j) Uncertainty and traceability requirements,

(k) Neutron self shielding, and

(l) Availability, transportability, cost, handling and storage risks, and practicality.

5.8.1.1 Sometimes representative standards may not be available for calibration. In such cases calculated correction factors may be applied to generic calibration standards to allow for the difference(s). The calibration range may be extended by calculation for one or more parameters. Similarly calculation is often used to assess the uncertainty associated with the calibrations. The calculation may use established radiation transport codes that have been validated and verified for similar uses. The calculations should be documented sufficiently to allow replication. This should be performed by suitably qualified and experienced personnel and reviewed by a peer member. For example, ^{252}Cf can be used to simulate $^{240}Pu_{eff}$; calculation can correct compact SNM items for self-multiplication and gamma ray self shielding.

5.8.2 Total calibration uncertainty should be determined as a part of the calibration process. Calibration uncertainty is then included in propagated uncertainty as a bias. Uncertainty in standard values, uncertainty of calibration measurements because of counting statistics, uncertainty from fitting calibration curves, and other parameters affect the total calibration uncertainty.

5.8.3 Calibration validation, or confirmation, may be performed to ensure that the calibration accurately reflects the response of the measurement instrumentation to radiation of interest. This can sometimes be conducted as a part of the measurement control program. Depending on regulatory requirements, the validation may be conducted using standards or process materials that are not traceable to a national measurement base, but whose radionuclide content is well known. Measured values for these items must agree within stated measurement uncertainty to validate the calibration. The validation requirements for a new measurement technique should be more rigorous than for a mature measurement method. Calibration validation typically includes measurement of actual process items. Parameters important to the assay method should, where practical, be varied to ensure that the calibration is valid over the range of expected values for each parameter.

5.8.4 Calibration activities need to be documented. Documentation should include sufficient information to reconstruct each calibration for each instrument. Documentation should include the calibration procedure, calibration measurement results, traceability of standards used, and other information deemed important to the calibration activities by measurement personnel.

5.9 *Operation*:

5.9.1 A measurement procedure is needed for each NDA technique. The measurement procedure should describe the steps required to perform measurements of items of unknown content. Operational procedures typically describe administrative responsibilities for staffing, oversight of measurements, and performance of measurements. Any safety precautions are usually noted in measurement procedures. Materials needed to conduct the measurements are listed. Procedures also are used to define item acceptance criteria (that is, describe the characteristics of items for which the technique is capable of providing accurate measurement results). Measurement control requirements and procedures for performing measurements in support of the measurement control program are also described. Reporting and data storage requirements are also typically included in measurement procedures. Developing a procedure for an analytical method is not an adequate substitute for expertise of the technical personnel involved.

5.9.2 Training of measurement personnel is required. The level of training needed is dependent upon the complexity of the measurement technique and the responsibilities of the personnel. Guides C 986 and C 1490 include extensive guidance regarding training programs.

5.9.3 Training requirements often extend beyond measurement personnel. Obtaining the best results from NDA techniques require training of personnel who package items for measurement, install and maintain measurement instrumentation, perform instrument calibrations, perform measurements, and interpret measurement results.

5.9.4 Analysis of data obtained from NDA measurements is required to convert counting information to the desired results, typically mass or activity of radionuclides contained in each measured item. Depending on the assay technique used, the measurement instrumentation and software available, the result may be provided automatically or a significant amount of data processing by qualified professionals may be required.

5.9.5 Reviews of data analysis methods should be conducted by qualified professionals for all measurement techniques. Expert review software may be used to perform part of the review for individual items. Administrative reviews typically include checks to ensure that items are correctly identified, values have been correctly entered, measurement control has been properly established and verified, and that all applicable procedures have been followed. In addition, expert technical reviews are conducted to ensure the appropriateness of the assay technique for the items measured and to review the raw data and measurement results for potential problems. Final results should not be reported until all necessary reviews have been completed.

5.10 *Quality Control*:

5.10.1 A measurement control program shall be established for all measurement systems. The purpose of a measurement control program is to demonstrate that a measurement process produces measured values of the required quality over the period of time the process is operating. The measurement control program also indicates if the measurement system performance has changed relative to its performance during calibration and operational verification. Precision, accuracy, total uncertainty, or minimum detectable quantity may represent the quality of a measurement. Further discussion on precision, accuracy, and total uncertainty is contained in 5.10.

5.10.2 Conditions unique to measurement control assessment of NDA instrumentation exist. Because NDA instrumentation measures radioactive materials, the values of sources used to track instrument performance do not remain constant over time and, in many cases, change considerably with time. This is commonly accounted for by decay correcting the results of measurement control data to a common date for a given

instrument or measurement control source. In addition, decay of measurement control sources may result in worsening of precision and accuracy (bias) for measurement of that source by the instrumentation monitored in the measurement control program. This is expected and should be accounted for by control evaluation methods.

5.10.3 In some cases (for example, gamma ray spectroscopy), multiple parameters are available from each measurement that can and should be used to evaluate the condition of measurement instrumentation. Using gamma ray spectroscopy as an example, an evaluation of the quality of each measurement (not only measurements of well controlled sources) can be achieved by monitoring parameters such as the full width at half maximum (FWHM) and peak shape for gamma ray peaks at specific energies, energy calibration, and system dead time. NDA subject matter experts should be consulted for each measurement application to determine the best methods for use in assessing measurement control.

5.10.4 In general, the quality of the measurements is monitored by periodically measuring a designated measurement control standard and comparing the measured value to an expected range of values. If the measured value of the standard falls within the expected range, the measurement process is said to be in control. If the measured value falls outside the expected range, it is an indicator of a potential problem with the measurement process. Potential problems must be investigated and resolved to ensure that the measurements being made are of the required quality. The measurements of the control standard and the expected range of values are evaluated using a valid statistical technique.

5.10.5 Multiple standards may be used to demonstrate the quality of measurements across a range of values. Separate comparisons may be made for each measurement control standard or they may be combined into a single comparison. Additionally, measurement system software may be programmed to automatically perform certain measurement control checks.

5.10.6 A measurement control standard need not be a certified reference material, but should have a well-established measurement history and provide a stable decay corrected result. The expected range of values of the standard can be derived from estimates of the measurement uncertainty and bias and is usually chosen to represent an interval that spans the range of expected measurements with 95 % or 99 % confidence. The measurement uncertainty includes the precision, but may include other sources of measurement variability. This is discussed further in the next section.

5.10.7 *Total Measurement Uncertainty (TMU) Analysis*—When a new measurement process is being adopted or an existing measurement process is being significantly altered, the quality of the measurements produced by the process should be evaluated. Typically, the quality of a measurement process is represented by the precision and accuracy (or bias) of the measurement. Definitions of these terms are provided below. These quantities are used when reporting measurement results to provide an indication of the quality of the measurement result. They are also used in establishing a measurement control program to monitor the quality of the measurement process over time. Additionally they may be used to demonstrate the process is meeting stated measurement performance objectives, which are bounds on the quality required of the measurement process. Typically, performance objectives are specified on the precision and accuracy of the measurement process. In the arena of NDA of nuclear materials, performance measures are required by regulation and accepted target values are available; see for example **(3-6)** and Chapter 23 of **(2)**. Before establishing a new measurement process, a review of all applicable regulations accepted target values and other factors influencing performance objective should be undertaken. Total measurement uncertainty analysis should also take into account all significant factors affecting both precision and accuracy. It should be noted that for NDA measurements, precision and accuracy can be radically different for measured items than for measurement control sources. Parameters that affect TMU, such as inhomogeneity, matrix effects, and exact measurement geometry are usually characterized very well for measurement control sources and not well known for measurement of items. These differences must be understood so that realistic estimates of TMU can be obtained.

5.10.7.1 The precision of a measurement process is the intrinsic variability of the process when those factors known to affect the measurement results are held constant. Precision can also be described as the repeatability of the measurement process. A full definition of repeatability can be found in Guide C 1215 or in **(7)** or **(8)**. Precision should be determined by performing replicate measurements of an item or items under normal measurement conditions. The estimated precision determined from counting statistics for neutron or gamma ray measurements cannot be arbitrarily substituted for the total random error determined from an item measurement. Under certain conditions, counting statistics are used to estimate instrument measurement precision (for example, gamma ray based isotopic composition estimates) for a single item measurement, but these estimates need to be validated.

5.10.7.2 Accuracy usually refers to the bias of the measurement process, which is a constant error between a measurement result and the accepted value of a measured quantity. Determination of bias requires the use of standards having a well-established value of the measured quantity; this value is referred to as the accepted reference value. Further details on the establishment and specification of precision and bias can be found in Guides C 1215, E 177, E 1323, and E 1488.

5.10.8 The careful identification and estimation of all significant factors affecting instrument response is important for measurement control purposes because measurement control programs track the performance of a measurement process over time. Failure to appropriately estimate all sources of variability can lead to frequent indications that the measurement process is not in control.

5.10.9 Validation of software used to produce calibrations and measurement results is a part of a comprehensive quality assurance program. Software shall be validated prior to use. Validation should include tests to ensure that the software produces correct results for all measurement and analysis situations that are anticipated during routine use.

6. Sources of NDA Error

6.1 Typical sources of error encountered in NDA measurements are described in the following sections together with means to eliminate or reduce their effects. These sources of error are common to calibration and assay of inventory items. They must be considered during calibration in order to obtain accurate assay results. The error sources discussed here are common to most NDA measurement techniques. Problem areas for specific techniques are treated in separate ASTM standards specific to those techniques. This discussion is divided into: *(1)* errors arising from measurement to measurement variability, which include those sources of error that can affect the observed response in repeated measurements of the same item, *(2)* errors because of item to item variability, which encompass additional sources of error that can alter the observed response per unit mass of the isotope(s) of interest in different items, and *(3)* the calibration error, which considers the effect on assay results of uncertainty in instrument calibration.

6.1.1 *Error from Measurement to Measurement Variability*—These sources of error can affect the observed response in repeated measurements of the same item.

6.1.1.1 *Counting Errors*—Counting experiments are characterized by an inherent variability that can be estimated for a given measurement process. The contribution to the total (relative) error because of counting can be reduced by increasing the total number of counts observed by: *(1)* counting for a longer time period, *(2)* increasing the detection efficiency through the use of additional detectors or reducing the item to detector distance, or *(3)* increasing the source strength in active systems.

6.1.2 *Background Contribution*—The measured response in NDA instrumentation contains some or all of the common background components discussed below. Additional sources of background contribution may also need to be addressed as sources of error for specific measurements systems and measurement scenarios.

6.1.2.1 *Instrument Background*—All electronic systems generate random noise that frequently determines the low signal limit to the sensitivity of the instrument.

6.1.2.2 *Ambient Background*—Cosmic rays and radiation from nearby accelerators, reactors, or radioactive sources may be detected by the instrument and erroneously attributed to the item being assayed. A common source of this problem relates to items temporarily placed near the system that are awaiting measurement or for which measurement has been completed. The ambient radiation often varies during routine operation. Therefore, where practical, the system should have sufficient shielding to be insensitive to ambient background radiation. In addition, the observed gross response should be corrected by subtracting the current measured background. Background measurements should be made sufficiently often that background variations will not significantly affect assay results. In addition, it is possible that the presence of an item in the instrument can alter the measured ambient or source backgrounds. When this is a problem, a mock item containing no fissionable or radioactive material should be used to determine the magnitude of the effect.

6.1.2.3 *Source Background*—Assay systems often include a radioactive source that will produce a background in the instrument. This background will be time dependent because of source decay.

6.1.2.4 *Induced Source Activity*—Active assay systems may induce a sufficient number of fissions in the item to produce a residual radioactivity that will interfere with a subsequent reassay within a short time period. This is of particular concern during calibration when a given calibration standard may be repeatedly measured in order to determine its mean response. In addition, the measured item can generate neutrons through interaction with cosmic rays that is a potential source of error for passive neutron measurement systems.

6.1.3 *Cross Coupling*—In instruments capable of simultaneously measuring more than one item in different chambers, the response in one chamber may be sensitive to the content of the other chambers. When this is a problem, the system should be calibrated and used with identical loading patterns. The magnitude and constancy of the abovementioned backgrounds should be determined prior to calibration and throughout the use of any NDA system. In some applications, a large background from the source can be used to monitor system performance, especially if the background energy spectrum is similar to that of the signal.

6.1.4 *Instrument Design*—Errors attributable to the design or operation of the system may arise from the effects described below.

6.1.4.1 *Resolution*—The measurement method and the specific instrument used must be able to resolve the desired response from interfering radiations caused by background or extraneous materials present in the item being assayed.

6.1.4.2 *Component Aging*—The operating characteristics of electronic and mechanical components change in time, creating shifts in the performance of the instrument.

6.1.4.3 *Response Profile*—The instrument views a given volume into which items are positioned for measurement. A measurement error may result if the response is not uniform throughout that volume because of nonuniform detection geometry or a nonuniform field of incident radiation (energy and intensity) in the case of active systems. This effect should be minimized through instrument design, incorporating if necessary the capability to scan and rotate the item being measured. With proper control of item position and item to standard similarity, residual effects can be included in the calibration.

6.1.5 *Assay Time Control*—An assay is often referenced to a fixed counting time period. If the time period fluctuates for any reason, the assay result is directly affected. Variations in assay time may occur if actuating switches change in position or operating characteristics, or if a timing mechanism is sensitive to power or temperature fluctuations. In a system based on count rate, the assay may not be affected if the time period is carefully measured.

6.1.5.1 A calorimetric assay must be allowed to reach thermal equilibrium before the measurement is stopped. An exception to this is when a software estimate of the equilibrium value is made. In this case the measurement can be stopped

before equilibrium is reached, however, the measurement precision may be affected.

6.1.6 *Instrument Environment*—A variety of environmental factors can affect the operation of an NDA system and introduce error into the assay result. If the assay system is sensitive to changes in operating environment, the instrument should be calibrated and used under the same environmental conditions. Typical effects include those described below.

6.1.6.1 *Temperature*—Detection equipment often displays sensitivity to fluctuations in the ambient temperature. Also, in active assay systems incorporating a well moderated interrogating neutron spectrum, changes in the moderator density because of temperature variations may produce changes in the neutron energy spectrum. If such effects are present, the system should be operated in a controlled temperature environment to limit the range of variation. If this is not possible and the diurnal or seasonal effect is significant, the measured response of assay items can be correlated with the ambient temperature at the time of measurement. A correction to each subsequent measurement then should be made on the basis of the resulting temperature dependent function. In this case, the temperature of the critical environment (for example, moderator) at the time of measurement must be recorded.

(1) Increases in the fluctuation of room temperature may increase the variability of calorimeter baseline results.

6.1.6.2 *Humidity*—High voltage leakage and the tendency for arcing increase at high humidity. The effect may be either a gradual change or a sudden shift in instrument performance. The effect can be minimized by proper equipment design and controlled by removing excess moisture from the air.

6.1.6.3 *Electrical Power*—Fluctuations in the electrical supply used to power an instrument may affect its operation. Line voltage or frequency changes and spurious electrical noise spikes coupled through the power line, ground loops, or broadcast to the instrument may have a significant effect on its operation. For this reason, instruments, if necessary, should be powered with regulated, filtered lines or by battery. Instruments should be properly grounded and shielded, and should be located in areas physically removed from electrical noise generating machinery unless insensitivity to such noise has been demonstrated.

6.1.7 *Errors from Item-to-Item Variability*—If the response of the instrument is affected by any property of the item container or its contents other than the mass of the isotope(s) of interest, error can be introduced into the assay. The sensitivity of the measured response must be investigated for the possible item to item differences discussed below.

6.1.7.1 *Containers*—Materials containing radionuclides are usually placed into containers for storage, handling, and measurement. Container dimensions, structural composition, and packaging procedures should be developed to reduce container to container differences and thereby minimize any errors attributable to this cause. The instrument should be designed to be as insensitive as possible to container differences.

6.1.7.2 *Radionuclide Distribution*—If the instrument has a nonuniform response at various locations within its detection chamber, the measured response will depend on the distribution of the radionuclide within its container. Scanning and rotating the item may help to reduce the resultant error.

6.1.7.3 *Self-Attenuation*—Distribution of the isotope(s) of interest in particles or lumps can cause error in the measured response because of self absorption of emitted radiations and self shielding of incident gamma rays or neutrons in active systems. If particle size is uniform and well controlled, calibration standards will eliminate this problem.

6.1.7.4 *Isotopic Composition*—The isotopes of uranium and plutonium and their radioactive decay products are encountered in variable proportions. If the measured response is sensitive to radioisotopes other than the isotope(s) of interest, the nuclidic composition of items being assayed must be known and should be identical to that of the standards.

6.1.7.5 *Matrix Effects*—The penetrability of incident or emerging radiations may be significantly affected by the presence and distribution of materials that comprise the matrix within which the radionuclides of interest are embedded. The probability that these reactions will occur changes as the atomic number and density of the matrix material increase. In general, passive neutron measurements are less susceptible to matrix effects than active neutron measurements or gamma ray measurements. Corrections can generally be employed to reduce or eliminate these effects. For gamma ray assay, such corrections are typically based on the observed intensity of two or more gamma rays of different energy emitted by the same radionuclide, or on the transmission of a gamma rays emitted by an external source. For neutron assay, incident or emerging neutrons can lose energy, be parasitically absorbed and thus lost from the system, or be absorbed in fissile nuclei and induce those nuclei to fission (multiplication). Matrix material may result in either an increase or a decrease in the observed neutron response. When hydrogenous or other moderating materials comprise the matrix, neutrons are moderated to low energies, thereby increasing their probability for subsequent absorptive reactions. Content limits must be established for separate categories if these effects are severe. A correction sometimes can be effected through the use of an appropriate spectral indicator (for example, a thermal neutron flux probe).

6.1.8 *Calibration Error*—The error in the calibration curve arises in part from random errors associated with the measurement process and the errors associated with the known values of the standards. Inexact response models can introduce additional calibration errors.

6.1.8.1 Short term fluctuations in instrument operation or environment can be absorbed in the calibration by collecting calibration data over a period that includes the range of fluctuation to be encountered in normal operation. The resulting dispersion in the calibration data will tend to reflect the variability in the measured response because of operational or environmental fluctuations. The estimated uncertainty in the fitting parameters will, in turn, reflect this dispersion in the calibration data.

6.1.8.2 Item to item differences that perturb the observed response for a given mass of the nuclide(s) of interest can be included in the calibration by selecting a large number of standards and ensuring that the range of the perturbing property in the standards reflects the range to be encountered in the

unknown items. While this technique may be an acceptable (and perhaps the only) means of treating normal manufacturing tolerances in product material (for example, cladding thickness of reactor fuel pins), it is not recommended for large effects in other types of material. When such perturbing effects are included in the calibration, the contribution from the uncertainty in the calibration curve to the estimated uncertainty in an assay result necessarily will be larger than when the perturbing property is measured for each item and a correction is made to the observed response.

6.1.8.3 Uncertainty in the radionuclide content of the calibration standards used will affect the calibration uncertainty.

7. Test Methods

7.1 *ASTM Standards*:

7.1.1 C 986 Guide for Developing Training Programs in the Nuclear Fuel Cycle

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

7.1.3 C 1068 Guide for Qualification of Measurement Methods by a Laboratory within the Nuclear Industry

7.1.4 C 1128 Guide for Preparation of Working Reference Materials for Use in the Analysis of Nuclear Fuel Cycle Materials

7.1.5 C 1156 Guide for Establishing Calibration for a Measurement Method Used to Analyze Nuclear Fuel Cycle Materials

7.1.6 C 1210 Guide for Establishing a Measurement System Quality Control Program

7.1.7 C 1297 Guide for Laboratory Analysts for the Analysis of Nuclear Fuel Cycle Materials

7.1.8 E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

7.1.9 E 181 Test Methods for Detector Calibration and Analysis of Radionuclides

7.1.10 E 691 Practice for Conduction an Interlaboratory Study to Determine the Precision of a Test Method

7.1.11 E 1323 Guide for Evaluating Laboratory Measurement Practices and the Statistical Analysis of the Resulting Data

7.1.12 E 1488 Guide for Statistical Procedures to Use in Developing and Applying ASTM Test Methods

7.2 *ANSI Standards*:

7.2.1 N15.5 Statistical Terminology and Notation for Nuclear Materials Management

7.2.2 N5.36 NDA Measurement Control and Assurance

7.3 *Other*:

7.3.1 Chapman, J., "Waste Characterization and Assay Overview," April 7, 1997

7.3.2 Bruckner, L. A., Hume, W. H., and Delvin, W. L., "On Precision and Accuracy (Bias) Statements for Measurement Procedures," LA-11190-MS

8. Measurement Documentation

8.1 It is good practice to have several types of documentation available for review. The documents describe the procedure chosen for analysis, data to show that the instrument was functioning properly, and reports that give information showing how the result for the measured items was obtained.

8.2 *Measurement Description*—Provide the basic philosophy used to select the measurement method. Describe the calibration method used to convert detector response to the radionuclide activity, concentration, or mass. Show the major algorithms used to obtain the activity from the response. Reference the measurement procedure used to obtain the data.

8.3 *Detector Calibration and Quality Assurance Information*—Provide calibration data for each instrument used to quantify the activity, concentration or mass of the radionuclide(s). In addition, provide control charts that indicate the instrument was functioning within expected statistical limits at the time of the measurement.

8.4 *Complete Report of Measured Items*—Include all information used to obtain the results. This should include raw data should another analysis be necessary. This information should include the name of the individual acquiring the data, date of acquisition, file name containing the raw data, calibration information, peak areas (if appropriate), and correction factors. This report provides information necessary to review the results by NDA professionals or auditors. The data may be stored as hard copy or electronic (data file).

8.5 *Summary Report*—This report is a brief summary of information that needs to be conveyed to the customer or manager. This report usually contains the item identification and reported activity, concentration or mass of the radionuclide(s) of interest in conjunction with additional contract/facility specific information. A reference should be included in this summary report that references the information included in 8.3 should a more detailed review be necessary.

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(**1**) Reilly, D., Ensslin, N., Smith, H., Kreiner, S., *Passive Nondestructive Assay of Nuclear Materials*, United States Nuclear Regulatory Commission, NUREG/CR 5550, 1991.

(**2**) Gozani, T., *Active Nondestructive Assay of Nuclear Materials*, United States Nuclear Regulatory Commission, NUREG/CR 0602, 1981.

(**3**) Guardini, S., "ESARDA Performance Values of Nondestructive Assay Techniques Applied in Safeguards," *Transactions of the American Nuclear Society 4th International Conference of Facility Operations-Safeguards Interface*, Vol 43, Supplement 1, 1991, pp. 66-67.

(**4**) Hsue, S. T., and Sampson, T. E., "Non-destructive Assay Techniques and Associated Measurement Uncertainties," *Journal of the Institute of Nuclear Materials Management*, XXI, 1992, pp. 17-25.

(**5**) Mitchell, W. G., "Measurement Technology at Selected DOE Facilities: A Status Report," *Journal of the Institute of Nuclear Materials Management*, XXI, 1992, pp. 32-42.

(**6**) Ensslin, N., Langner, D. G., Menlove, H. O., Miller, M. C., and Russo, P. A., "Some Target Assay Uncertainties for Passive Neutron Coincidence Counting," *Journal of the Institute of Nuclear Material Management*, XIX, 1990, pp. 610-614.

(**7**) International Organization for Standardization, Guide to the Expression of Uncertainty in Measurement, 1993.

(**8**) Taylor, B. N., and Kuyatt, C. E., Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results, 1994, National Institute of Standards and Technology.

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