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Standard Guide for Making Quality Nondestructive Assay Measurements[1]

This standard is issued under the fixed designation C1592/C1592M; 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 Quality Measurement Practices for performing measurements of radioactive material using nondestructive assay (NDA) instruments. The primary purpose of the guide is to assist 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 guidelines to achieving quality NDA measurements 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 or atomic radiations, including photons, neutrons, or 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 C1490. This guide is to be used as a reference, and to supplement the critical thinking, professional skill, expert judgment, 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.

1.5 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system are not necessarily exact equivalents; therefore, to ensure conformance with the standard, each system shall be used independently of the other, and values from the two systems shall not be combined.

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

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

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C986 Guide for Developing Training Programs in the Nuclear Fuel Cycle (Withdrawn 2001)[3]

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

C1030 Test Method for Determination of Plutonium Isotopic Composition by Gamma-Ray Spectrometry

C1068 Guide for Qualification of Measurement Methods by a Laboratory Within the Nuclear Industry

C1128 Guide for Preparation of Working Reference Materials for Use in Analysis of Nuclear Fuel Cycle Materials

C1133/C1133M Test Method for Nondestructive Assay of Special Nuclear Material in Low-Density Scrap and Waste by Segmented Passive Gamma-Ray Scanning

C1156 Guide for Establishing Calibration for a Measurement Method Used to Analyze Nuclear Fuel Cycle Materials

C1207 Test Method for Nondestructive Assay of Plutonium in Scrap and Waste by Passive Neutron Coincidence Counting

[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.

[3] The last approved version of this historical standard is referenced on www.astm.org.

C1210 Guide for Establishing a Measurement System Quality Control Program for Analytical Chemistry Laboratories Within Nuclear Industry
C1215 Guide for Preparing and Interpreting Precision and Bias Statements in Test Method Standards Used in the Nuclear Industry
C1221 Test Method for Nondestructive Analysis of Special Nuclear Materials in Homogeneous Solutions by Gamma-Ray Spectrometry
C1297 Guide for Qualification of Laboratory Analysts for the Analysis of Nuclear Fuel Cycle Materials
C1254 Test Method for Determination of Uranium in Mineral Acids by X-Ray Fluorescence
C1268 Test Method for Quantitative Determination of ^{241}Am in Plutonium by Gamma-Ray Spectrometry
C1316 Test Method for Nondestructive Assay of Nuclear Material in Scrap and Waste by Passive-Active Neutron Counting Using ^{252}Cf Shuffler
C1455 Test Method for Nondestructive Assay of Special Nuclear Material Holdup Using Gamma-Ray Spectroscopic Methods
C1458 Test Method for Nondestructive Assay of Plutonium, Tritium and ^{241}Am by Calorimetric Assay
C1490 Guide for the Selection, Training and Qualification of Nondestructive Assay (NDA) Personnel
C1493 Test Method for Non-Destructive Assay of Nuclear Material in Waste by Passive and Active Neutron Counting Using a Differential Die-Away System
C1500 Test Method for Nondestructive Assay of Plutonium by Passive Neutron Multiplicity Counting
C1514 Test Method for Measurement of ^{235}U Fraction Using Enrichment Meter Principle
C1592 Guide for Making Quality Nondestructive Assay Measurements
C1673 Terminology of C26.10 Nondestructive Assay Methods
C1718 Test Method for Nondestructive Assay of Radioactive Material by Tomographic Gamma Scanning
C1726/C1726M Guide for Use of Modeling for Passive Gamma Measurements
C1807 Guide for Nondestructive Assay of Special Nuclear Material (SNM) Holdup Using Passive Neutron Measurement Methods
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E181 Test Methods for Detector Calibration and Analysis of Radionuclides
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
E1323 Guide for Evaluating Laboratory Measurement Practices and the Statistical Analysis of the Resulting Data
E1488 Guide for Statistical Procedures to Use in Developing and Applying Test Methods

2.2 *ANSI Standards:*[4]

ANSI N15.36 Methods Of Nuclear Material Control - Measurement Control Program - Nondestructive Assay Measurement Control And Assurance
ANSI N15.5 Statistical Terminology and Notation for Nuclear Materials Management

2.3 *Other Documents:*

ESARDA NDA Good Practices Guide[5]
NPL Good Practices Guide[6]

3. Terminology

3.1 Definitions presented here are confined to those terms not defined in Terminology C1673, other common nuclear materials glossaries/references or whose use is specific to this application.

3.2 *Definitions:*

3.2.1 *differential die away technique (DDT), n*—also referred to as DDA, an NDA technique for characterizing fissionable material in scrap and waste using prompt neutrons from fissions induced by neutron generator interrogation source.

3.2.2 *in-process material, n*—the nuclear material in a process stream, excluding holdup.

3.2.3 *passive neutron coincidence counting, n*—a technique used to measure the rate of temporally coincident neutron emission in the assay item.

3.2.4 *Poisson assumption, n*—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 statistical uncertainty.

3.2.5 *procedure, n*—a set of systematic instructions for using a method of measurement or of the steps associated with the method.

3.2.6 *qualitative analysis, n*—an analysis or measurement in which some or all of the attributes or characteristics of an item are determined, but no quantitative estimates of the radionuclides are made.

3.2.7 *quality measurement practice, n*—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).

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

3.2.9 *replicate, n*—one of several identical experiments, procedures, or samples; it is the general case for which duplicate and triplicate, consisting of two and three measurements, respectively, are the special cases.

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

[5] Available from ESARDA Secretariat c/o European Commission JRC Bldg. 42A Via E. Fermi, 2749 21027-Ispra (VA), Italy, https://esarda.jrc.ec.europa.eu.

[6] Available from National Physical Laboratory, Hampton Road, Teddington, Middlesex, TW11 0LW, https://www.npl.co.uk.

3.2.10 *segmented gamma scanner, n*—an NDA 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.

3.2.11 *shift-register-based coincidence circuit, n*—a dedicated electronic circuit for measuring temporally correlated quantitities relevant to passive neutron coincidence counting.

3.2.12 *shuffler, n*—an NDA technique for characterizing the delayed neutrons from fissionable nuclides in scrap and waste using delayed neutrons induced by ^{252}Cf interrogation source.

3.2.13 *verification, n*—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.

4. Significance and Use

4.1 NDA measurement practices aimed at achieving quality results 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 or require 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**).[7] A partial listing of measurement methods and applicable use references is provided in 5.5.1. 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, geometry, thickness, and distribution of fissile material, 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, maximizing precision and minimizing bias) can significantly depend on the degree of adherence to this waste management plan.

4.7 This guide addresses elements of quality measurement 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.

5. Quality Measurement 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. The primary goal of NDA measurements is to arrive at a quality result that satisfies the user's measurement needs without the necessity to alter the item. Adequately analyzing problems and applying appropriate measurement techniques support this goal.

5.2 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.

5.3 The observed response of an NDA system shows sensitivity to a wide variety of factors that can bias the assay result.

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

By careful selection of the measurement technique, attention to potential sources of uncertainty, 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.

5.4 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 parameters that should be considered when selecting NDA measurement systems are listed below, not necessarily in the order of priority:

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 chemical form (for example, oxide, fluoride etc.),

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

e) The container and packing material (for example, size, wall thickness, mass, wall material, etc.),

f) Environmental conditions,

g) Measurement quality objectives,

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

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

j) Background radiation,

k) Costs (instrument, set up, personnel, and operating costs),

l) Availability of instrumentation,

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

n) Training requirements,

o) Ease of operation,

p) Program schedule,

q) Surface dose rate, and

r) Item throughput.

5.5 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 nuclide(s) of the elemental species and, in some cases, by their decay products requires knowledge of the relative radionuclide composition of the item assayed.

5.5.1 Many of the approaches to specific NDA measurement techniques are described by ASTM Standards as shown in Table 1. A list of applicable ASTM and ANSI standards are also provided in Table 1. Other standards may also be considered.

5.5.2 *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 from an external neutron source. The quantity of a particular nuclide 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. Important considerations for making quality neutron measurements include:

a) Elements that initiate (α, *n*) interferences,

b) Hydrogen content,

c) Neutron moderation and absorption (poisons),

d) Container wall effects,

e) Influence of uranium on plutonium assay or of plutonium on uranium assay,

f) Source self-shielding,

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

h) Unexpected sources of neutrons,

i) Chemical composition,

j) System dead-time,

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

l) Measurement geometry,

m) Background radiation (for example, spallation neutrons),

n) Density,

o) Neutron multiplication,

p) Delayed neutron emissions from fission or nuclear reactions,

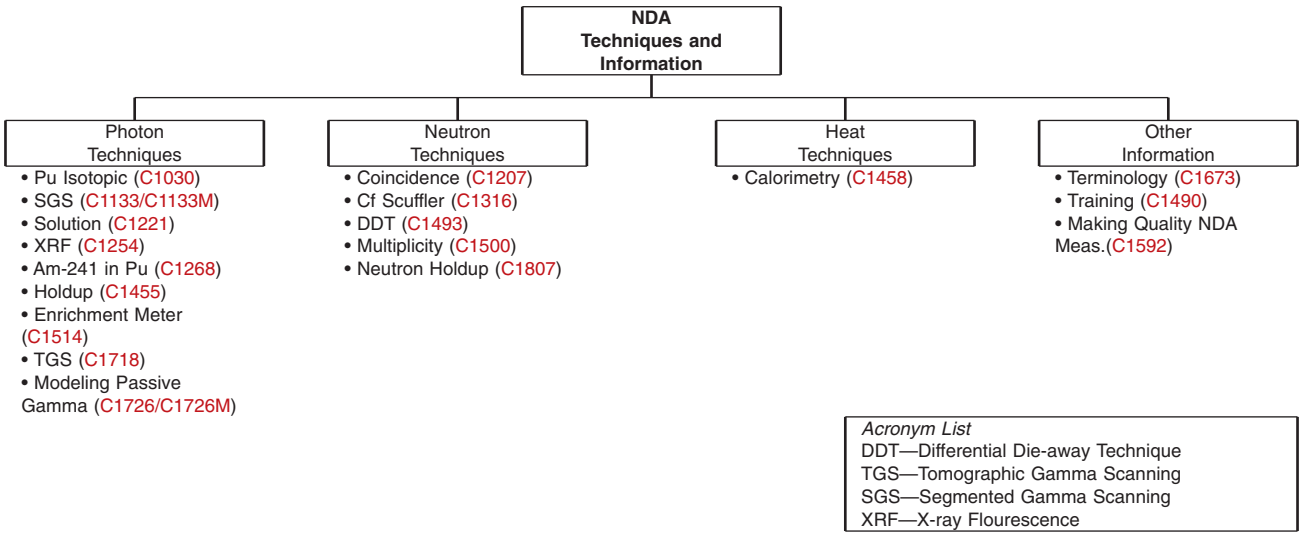
q) (γ, n) emissions, perhaps resulting from (n, γ) reactions or gamma-rays, (n, 2n) reactions, and

r) Buildup of ^{250}Cf in aging ^{252}Cf sources used for calibration.

5.5.2.1 *Passive Neutron Counting*—The primary strength of passive neutron counting is 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 passive neutron emission measurement programs are often considerably less than for active neutron measurement techniques. In addition, because external neutron sources are not required, risk of personnel exposure to radiation is generally lower for passive neutron assay. The primary disadvantages of passive neutron assay relative to other neutron assay methods are that counting rates are often lower and contaminants (for example, (α, *n*) neutrons, presence of poisons) might influence the total and coincident neutron count rate resulting in a bias.

(1) Total Neutron Counting is most suitable 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 nuclide ratios are well known. When the measured items do not have all of these

TABLE 1 ASTM Standards Related to NDA Techniques



Applicable ASTM Standards:
Guide C986—Standard Guide for Developing Training Programs in the Nuclear Fuel Cycle
Guide C1009—Standard Guide for Establishing a Quality Assurance Program for Analytical Chemistry Laboratories within the Nuclear Industry
Guide C1068—Standard Guide for Qualification of Measurement Methods by a Laboratory within the Nuclear Industry
Guide C1128—Standard Guide for Preparation of Working Reference Materials for Use in the Analysis of Nuclear Fuel Cycle Materials
Guide C1156—Standard Guide for Establishing Calibration for a Measurement Method Used to Analyze Nuclear Fuel Cycle Materials
Guide C1210—Standard Guide for Establishing a Measurement System Quality Control Program
Guide C1297—Standard Guide for Laboratory Analysts for the Analysis of Nuclear Fuel Cycle Materials
Test Method C1500—Standard Test Method for Nondestructive Assay of Plutonium by Passive Neutron Multiplicity Counting
Guide C1514—Standard Test Method for Measurement of U Fraction Using the Enrichment Meter Principle 235
Practice E177—Standard Practice for Use of the Terms Precision and Bias in ASTM Test Methods
Test Methods E181—Standard Test Methods for Detector Calibration and Analysis of Radionuclides
Practice E691—Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
Guide E1323—Standard Guide for Evaluating Laboratory Measurement Practices and the Statistical Analysis of the Resulting Data
Guide E1488—Standard Guide for Statistical Procedures to Use in Developing and Applying ASTM Test Methods

Applicable ANSI Standards:
ANSI N15.36—NDA Measurement Control and Assurance
ANSI N15.5—Statistical Terminology and Notation for Nuclear Materials Management

attributes, the user must be cautious with respect to sources of measurement uncertainty. 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 elemental mass. Coincidence neutron counting is less sensitive to many of the biases typical of total neutron counting (for example, the presence of [α, *n*] target material) because their effect is reduced. Spontaneous fission of ^{244}Cm and certain other nuclides 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 the impact of cosmic ray background even when the count rates for the higher moments are low.

5.5.2.2 *Active Neutron Interrogation* is applicable when ^{235}U is present or when passive signals are weak. Selection of an appropriate interrogating-neutron source 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.

(1) Thermal neutrons can be used for active neutron assay systems if they can adequately penetrate the item. Additional information may be required about the geometry, homogeneity, and the mass level of the fissile material to be assayed. The presence of thermal-neutron absorbers such as gadolinium (Gd) in light-water-reactor (LWR) fuel may preclude the use of a thermal neutron interrogation. Thermalneutron interrogation may be possible for small items with high moderation, (for example, hydrogen (H) content, 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. Active neutron counters

such as the Active Well Coincidence Counter (AWCC) which use thermal neutron based interrogation of uranium bearing samples, are widely used in nuclear safeguards and security applications.

(2) For the assay of fissile items that have poor penetrability for thermal neutrons, interrogation by neutrons having energies greater than thermal is recommended. Interrogating-neutron spectra can originate from various sources such as spontaneous fission nuclides, neutron generators or accelerators.

(3) 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 (for example, bare and cadmium covered neutron detectors), coincidence counting, timing, and shielding.

5.5.3 *Calorimetric Assay*—Applications of calorimetry to NDA refer to the measurement of heat flow generated from radioactive decay. Calorimetric assay typically provides very good precision and low bias. It is most often used to assay plutonium with ^{241}Am, and tritium. Typical assay times range from 4 to 24 hours. Typically calorimeter chambers are 0.203 m [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 actinide isotopic composition. For plutonium, this is typically accomplished using high-resolution gamma-ray spectrometry. Important considerations in making quality calorimetric assays include:

a) Heat-generating contaminants (for example, chemical curing, biological decay, bacterial reaction, and radiolysis),

b) Isotopic composition,

c) Chemical reactions that produce or consume heat,

d) Phase changes that produce or consume heat,

e) Item position,

f) Heat transfer characteristics of the item and its packaging, which might affect total measurement time and results, and

g) Drift in bridge potential (or baseline power for servo-control method).

5.5.4 *Photon Techniques* are based on the detection of gamma or X-rays that are emitted with discrete energies characteristic of specific nuclides or elements. The intensity of photons of a specific energy is related to the quantity or fraction of a particular nuclide or element. The relative intensity of gamma-rays from different radionuclides can be related to the relative abundance of those nuclides. There are a variety of detectors available, which generally span the range of efficiency and resolution from relatively high to low.

Important considerations for making quality photon measurements include:

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

b) Matrix heterogeneity, source geometry (for example, lumps) causing self-absorption,

c) Non-representative calibration standards,

d) Attenuation,

e) Shielding and collimation as appropriate,

f) Graded shielding to minimize interference of X-rays from higher Z shielding materials,

g) Low signal to noise ratio,

h) Signal distortion (for example, tailing, pulse pileup/random coincidence summing, true coincidence summing)

i) Dead-time correction,

j) Measurement geometry,

k) Item size (physical dimensions),

l) Container packaging and matrix attenuation,

m) Background radiation,

n) Interfering radiation, and

o) Decay of radioactive sources used to routinely test the stability/functionality of a measurement system, transmission sources, and rate-based correction sources.

5.5.4.1 *Isotopic Composition*—Gamma-ray spectrometry may be used to determine isotopic composition (Test Methods C1030, C1268). Isotopic composition from gamma-ray spectrometry is often used to support both calorimetry (Test Method C1458) and neutron techniques (Test Methods C1207, C1493, C1500, and C1316), as well as for other applications.

5.5.4.2 *Quantitative Assay*—Gamma-ray spectrometry is used for quantitative assay of specific nuclides 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. The assay geometry including the source-detector distance, shielding, collimator aperture, field of view should be optimized.

5.6 Specific radionuclides 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.

5.7 *Calibration*—Calibration provides a mathematical relationship to correlate detector response with characteristics of the measured item. In the context of photon-based NDA techniques, three main types of detector calibration can be distinguished: *(1)* Energy, *(2)* Peak shape, *(3)* Efficiency. Methods used for calibration are specific to a particular 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 measurement 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) Energy and Peak shape (FWHM) calibration for photon methods,

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

d) Measuring instrument repeatability,

e) Determining the sources and magnitude of bias, and

f) Determining total calibration uncertainty.

5.7.1 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 properties,

g) Emission rate for radiation of interest,

h) Number of standards required,

i) Replacement interval and accounting for standards with short half-life, chemical instability, or pressure build up,

j) Time-dependent isotopic composition,

k) Uncertainty and traceability requirements,

l) Neutron self-shielding, and

m) Availability, transportability, cost, handling and storage risks, and practicality.

5.7.1.1 Sometimes sufficiently representative standards may not be available for calibration. Mathematical modeling techniques can be used in such cases. However, the model-based calibrations must be validated using sufficiently representative measurements and properly documented. Calculated correction factors may be applied to generic calibration standards to allow for the difference(s) between calibration standards and items. 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. Examples include: ^{252}Cf can be used as surrogate to $^{240}Pu_{eff}$; modeling can correct results for items for neutron self-multiplication and gamma-ray self-shielding in items containing fissile materials.

5.7.2 Total calibration uncertainty should be determined as a part of the calibration process. Calibration uncertainty is then included in propagated uncertainty as a systematic uncertainty. 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.

5.7.3 Calibration verification and validation 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.

5.7.4 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, NDA personnel performing the measurement, and other information deemed important to the calibration activities by measurement personnel.

5.8 *Operation:*

5.8.1 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.*

5.8.2 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. Guide C1490 includes extensive guidance regarding training programs.

5.8.3 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. Managers who have oversight of NDA results must also be adequately trained.

5.8.4 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