



U.S. ATOMIC ENERGY COMMISSION

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DIRECTORATE OF REGULATORY STANDARDS

## REGULATORY GUIDE 5.11

### NONDESTRUCTIVE ASSAY OF SPECIAL NUCLEAR MATERIAL CONTAINED IN SCRAP AND WASTE

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# NONDESTRUCTIVE ASSAY OF SPECIAL NUCLEAR MATERIAL CONTAINED IN SCRAP AND WASTE

## A. INTRODUCTION

Section 70.51, "Material Balance, Inventory, and Records Requirements," of 10 CFR Part 70, "Special Nuclear Material," requires licensees authorized to possess at any one time more than one effective kilogram of special nuclear material to establish and maintain a system of control and accountability such that the limit of error of any material unaccounted for (MUF), ascertained as a result of a measured material balance, meets established minimum standards. The selection and proper application of an adequate measurement method for each of the material forms in the fuel cycle is essential for the maintenance of these standards.

With proper controls, licensees may select nondestructive assay (NDA) as an alternative to traditional measurement methods. This guide details procedures acceptable to the Regulatory staff to provide a framework for the utilization of NDA in the measurement of scrap and waste inventory components generated in conjunction with the processing of special nuclear materials (SNM). Subsequent guides will detail procedures specific to the application of a selected technique to a particular problem.

## B. DISCUSSION

### 1. Applicable Nondestructive Assay Principles

The nondestructive assay of the SNM content of heterogeneous material forms is achieved through observing either stimulated or spontaneously occurring radiations emitted from the isotopes of either plutonium or uranium, from their radioactive decay products, or from some combination of these materials. The isotopic composition must be known to permit a conversion of the amount of isotope measured to the amount of element present in the container. Assays are performed by isolating the container of interest to permit a measurement of its contents through a comparison with the response observed from known calibration standards. This technology permits quantitative assays of the SNM content of heterogeneous materials in short measurement times without sample preparation and without affecting the form of the material to be assayed. The proper application of this technology requires the understanding and control of factors influencing NDA measurements.

#### 1.1 Passive NDA Techniques

Passive NDA is based on observing spontaneously emitted radiations created through the radioactive decay of plutonium or uranium isotopes or of their radioactive daughters. Radiations attributable to alpha ( $\alpha$ ) particle

decay, to gamma ray transitions following  $\alpha$  and beta ( $\beta$ ) particle decay, and to spontaneous fission have served as the bases for practical passive NDA measurements.

#### 1.1.1 NDA Techniques Based on Alpha Particle Decay

Alpha particle decay is indirectly detected in calorimetry measurements. (Note: a small contribution is attributable to the  $\beta$  decay of  $^{241}\text{Pu}$  in plutonium calorimetry applications.) The kinetic energy of the emitted  $\alpha$  particle and the recoiling daughter nucleus is transformed into heat, together with some fraction of the gamma ray energies which may be emitted by the excited daughter nucleus in lowering its energy to a more stable nuclear configuration. The calorimetric measurement of the heat produced by a sample can be converted to the amount of  $\alpha$ -particle-emitting nuclides present through the use of the isotopic abundance and the specific power [watts  $\text{gm}^{-1} \text{sec}^{-1}$ ] of each nuclide.<sup>1</sup> Plutonium, because of its relatively high specific power, is amenable to calorimetry.

The interaction of high-energy  $\alpha$  particles with some light nuclides (e.g.,  $^7\text{Li}$ ,  $^9\text{Be}$ ,  $^{10}\text{Be}$ ,  $^{11}\text{Be}$ ,  $^{18}\text{O}$ , and  $^{19}\text{F}$ ) may produce a neutron. When the isotopic composition of the  $\alpha$ -particle-emitting nuclides is known and the content of high-yield ( $\alpha, n$ ) targets is fixed, the observation of the neutron yield from a sample can be converted to the amount of SNM present.

#### 1.1.2 NDA Techniques Based on Gamma Ray Analysis

The gamma ray transitions which reduce the excitation of a daughter nucleus following either  $\alpha$  or  $\beta$  particle emission from an isotope of SNM occur in discrete energies.<sup>2, 3</sup> The known  $\alpha$  particle decay activity of the SNM parent isotope and the probability that a specific gamma ray will be emitted following the  $\alpha$  particle decay can be used to convert the measurement of that gamma ray to a measurement of the amount of the SNM parent isotope present in the container being measured. High-resolution gamma ray spectroscopy is required when the gamma ray(s) being measured is observed in the presence of other gamma rays or X-rays which, without being resolved, would interfere with the measurement of the desired gamma ray.

#### 1.1.3 NDA Techniques Based on Spontaneous Fission

A fission event is accompanied by the emission of from 2 to 3.5 neutrons (depending on the parent nucleus) and an average of about 7.5 gamma rays. A

total of about 200 MeV of energy is released, distributed among the fission fragments, neutrons, gamma rays, beta particles, and neutrinos. Spontaneous fission occurs with sufficient frequency in  $^{238}\text{Pu}$ ,  $^{240}\text{Pu}$ ,  $^{242}\text{Pu}$ , and  $^{238}\text{U}$  to facilitate assay measurements through the observation of this reaction. Systems requiring the coincident observation of two or three of the prompt radiations associated with the spontaneous fission event provide the basis for available measurement systems.<sup>4</sup>

## 1.2 Active NDA Techniques

Active NDA is based on the observation of radiations (gamma rays or neutrons) which are emitted from the isotope under investigation when that isotope undergoes a transformation resulting from an interaction with stimulating radiation provided by an appropriate external source. Isotopic<sup>5</sup> and accelerator<sup>4</sup> sources of stimulating radiation have been investigated.

Stimulation with accelerator-generated high-energy neutrons or gamma rays should be considered only after all other NDA methods have been evaluated and found to be inadequate. Such systems have been tested to assay variable mixtures of fissile and fertile materials in large containers having a wide range of matrix variability. Operational requirements, including operator qualifications, maintenance, radiation shielding, and calibration considerations, normally require an inordinate level of support in comparison to the benefits of in-plant application.

Fission is readily induced by neutrons in the  $^{233}\text{U}$  and  $^{235}\text{U}$  isotopes of uranium and in the  $^{239}\text{Pu}$  and  $^{241}\text{Pu}$  isotopes of plutonium. Active NDA systems have been developed using spontaneous fission ( $^{252}\text{Cf}$ ) neutron sources, as well as ( $\gamma, n$ ) [Sb-Be] sources and a variety of ( $\alpha, n$ ) [Am-Li, Pu-Li, Pu-Be] sources.<sup>5</sup> In the assay of scrap and waste, the neutron-induced fission reactions are separated from background radiations through observing radiations above a predetermined energy level or through observing two or three of the radiations emitted in fission in coincidence.<sup>4</sup>

The detection of delayed neutrons or gamma rays has been employed using isotopic neutron sources to induce fission, then removing either source or container to observe the delayed emissions.

## 2. Factors Affecting the Response of NDA Systems

Regardless of the technique selected, the observed NDA response depends on (1) the operational characteristics of the system, (2) the isotopic composition of the SNM, (3) the amount and distribution of SNM, (4) the amount and distribution of other materials within the container, and (5) the composition and dimensions of the container itself. Each of these variables contributes to the overall uncertainty associated with an NDA measurement.

The observed NDA response represents primary contributions from the different SNM isotopes present in the container. To determine the amount of SNM present, the isotopic composition of the SNM must be known and the variation in the observed response as a function of varying isotopic composition must be understood. The effects due to items (3), (4), and (5) above on the observed response can be reduced through appropriate selection of containers, compatible segregation of scrap and waste categories, and consistent use of packaging procedures designed to improve the uniformity of container loadings.

### 2.1 Operational Characteristics

The operational characteristics of the NDA system, together with the ability of the system to resolve the desired response from a composite signal, determine the ultimate usefulness of the system. These operational characteristics include (1) operational stability, (2) geometric detection sensitivity, (3) stimulating radiation uniformity, and (4) energy of the stimulating radiation.

The impact of the operational characteristics noted above on the uncertainty of the measured response can be reduced through the design of the system and the use of radiation shielding (where required).

#### 2.1.1 Operational Stability

The ability of an NDA system to reproduce a given measurement may be sensitive to fluctuations in the operational environment. Temperature, humidity, and line voltage variations affect NDA systems to some extent. These effects may be manifested through the introduction of spurious electronic noise or changes in the high voltage applied to the detector(s) or amplifiers, thereby changing the detection efficiency. The environment can be controlled if such fluctuations result in severe NDA response variations which cannot be eliminated through calibration and operational procedures.

The sensitivity to background radiations can be monitored and controlled through proper location of the system and the utilization of radiation shielding, if required.

#### 2.1.2 Geometric Detection Sensitivity

The NDA system should be designed to have a uniform response throughout the detection chamber. The residual geometric response dependence can be measured using an appropriate source which emits radiation of the type being measured. The source should be small with respect to the dimensions of the detection chamber. The system response can then be measured with the source positioned in different locations to determine the volume of the detection chamber which can be reliably used.

An encapsulated Pu source can be used to test gamma ray spectroscopic systems, active or passive NDA systems detecting neutrons or gamma rays, or calorimetry systems. Active NDA systems can be operated in a passive mode (stimulating source removed) to evaluate the magnitude of this effect. Rotating and Scanning containers during assay is a recommended means of reducing the response uncertainties attributable to residual nonuniform geometric detection sensitivity.

### 2.1.3 Uniformity of Stimulating Radiation

The stimulating radiation field (i.e., interrogating neutron or gamma ray flux) in active NDA systems should be designed to be uniform in intensity and energy spectrum throughout the volume of the irradiation chamber. The residual effect can be measured using an SNM sample which is small with respect to the dimensions of the irradiation chamber. The response can then be measured with the SNM sample positioned in different locations within the irradiation chamber. If the same chamber is employed for irradiation and detection, a single test for the combined geometric nonuniformity is recommended.

Various methods have been investigated to reduce the response uncertainty attributable to a nonuniform stimulating radiation field, including rotating and scanning the container, source scanning, distributed sources, and combinations of these methods. Scanning a rotating container with the detector and source positions fixed appears to offer an advantage in response uniformity and is therefore recommended.

### 2.1.4 Energy of Stimulating Radiation

If the energy of the stimulating radiation is as high as practicable but below the threshold of any interfering reactions such as the neutron-induced fission in  $^{238}\text{U}$ , the penetration of the stimulating radiation will be enhanced throughout the volume of the irradiation chamber. A high-energy source providing neutrons above the energy of the fission threshold for a fertile constituent such as  $^{238}\text{U}$  or  $^{232}\text{Th}$  can be employed to assay the fertile content of a container.

The presence of extraneous materials, particularly those of low atomic number, lowers the energy spectrum of the interrogating neutron flux in active neutron NDA systems. Incorporating a thermal neutron detector to monitor this effect and thereby provide a basis for a correction to reduce the response uncertainty caused by this variable effect is recommended.

Active neutron NDA systems with the capability to moderate the interrogating neutron spectrum can provide increased assay sensitivity for samples containing small amounts of fissile material (<100 grams). This moderation capability should be removable to enhance the range of usefulness of the system.

## 2.2 Response Dependence on SNM Isotopic Composition

The observed NDA response may be a composite of contributions from more than a single isotope of uranium or plutonium. Observed effects are generally attributable to one of the three sources described below.

### 2.2.1 Multiple Gamma Ray Sources

Plutonium contains the isotopes  $^{238}\text{Pu}$  through  $^{242}\text{Pu}$  in varying quantities. With the exception of  $^{242}\text{Pu}$ , these isotopes emit many gamma rays.<sup>2,3</sup> The observed Pu gamma ray spectrum represents the contribution of all gamma rays from each isotope, together with the gamma rays emitted in the decay of  $^{241}\text{Am}$ , which may also be present.

Uranium gamma rays are generally lower in energy than Pu gamma rays. Uranium-232, occurring in combination with  $^{233}\text{U}$ , has a series of prolific gamma-ray-emitting daughter products which include  $^{228}\text{Th}$ , with the result that daughter products of  $^{232}\text{U}$  and  $^{232}\text{Th}$  are identical beyond  $^{228}\text{Th}$ .

### 2.2.2 Multiple Spontaneously Fissioning Pu Isotopes

In addition to the spontaneous fission observed from  $^{240}\text{Pu}$ , the minor isotopes  $^{238}\text{Pu}$  and  $^{242}\text{Pu}$  typically contribute a few percent to the total rate observed.<sup>6</sup> In mixtures of uranium and plutonium blended for reactor fuel applications, the spontaneous fission yield from  $^{238}\text{U}$  may approach one percent of the  $^{240}\text{Pu}$  yield.

### 2.2.3 Multiple Fissile Isotopes

In active systems, the observed fission response may consist of contributions from more than one isotope. For enriched uranium, if the energy spectrum of the stimulating radiation extends above the threshold for  $^{238}\text{U}$  fission, that response contribution will be in addition to the induced  $^{235}\text{U}$  fission response.

In plutonium, the observed response will be the sum of contributions from the variable content of  $^{239}\text{Pu}$  and  $^{241}\text{Pu}$ .

When elements (e.g., plutonium and uranium) are mixed for reactor utilization, the uncertainty in the response is compounded by introducing additional fissile components in variable combinations.

## 2.3 Response Dependence on Amount and Distribution of SNM in a Container

If a system has a geometrically uniform detection sensitivity and a uniform field of stimulating radiation (where applicable), a variation in the response per gram of the isotope(s) being measured is generally attributable to one of the three causes described below.

### 2.3.1 Self-Absorption of the Emitted Radiation Within the SNM

For a fixed amount of SNM in a container, the probability that radiation emitted by the SNM nuclei will interact with other SNM atoms increases as the localized density of the SNM increases within the container. This is a primary source of uncertainty in gamma ray spectroscopy applications. It becomes increasingly important as the SNM aggregates into lumps and is more pronounced for low-energy gamma rays.

### 2.3.2 Multiplication of Spontaneous or Induced Fission

The neutrons given off in either a spontaneous or an induced fission reaction can be absorbed in a fissile nucleus and subsequently induce that nucleus to fission, resulting in the emission of two or more neutrons. This multiplication results in an increased response from a given quantity of SNM. Multiplication affects the response of all active NDA systems and passive coincidence neutron or gamma ray detection systems used to observe spontaneous fission. This effect becomes increasingly pronounced as the energy of the neutrons traversing the container becomes lower or as the density of SNM increases within the container.

### 2.3.3 Self-Shielding of the Stimulating Radiation

This effect is particularly pronounced in active systems incorporating a neutron source to stimulate the fissile isotopes of the SNM to fission. More of the incident low-energy neutrons will be absorbed near the surface of a high-density lump of SNM, and fewer will penetrate deeper into the lump. Thus, the fissile nuclei located deep in the lump will not be stimulated to fission at the same rate as the fissile nuclei located near the surface, and a low assay content will be indicated. This effect is dependent on the energy spectrum of the incident neutrons and the density of fissile nuclei. It becomes increasingly pronounced as the energy of the incident neutrons is decreased or as the density of the SNM fissile content is increased. The density of fissile nuclei is increased when the SNM is lumped in aggregates or when the fissile enrichment of the SNM is increased.

### 2.4 Response Dependence on Amount and Distribution of Extraneous Materials within the Container

The presence of materials other than SNM within a container can affect the emitted radiations in passive and active NDA systems and can also affect the stimulating radiation in active assay systems. The presence of extraneous materials can result in either an increase or a decrease in the observed response.

Effects on the observed NDA response are generally attributable to one of the four causes described below.

### 2.4.1 Interfering Radiations

This problem arises when the material emits a radiation which cannot be separated from the desired signal. This problem is generally encountered in gamma ray spectroscopy and calorimetry applications as the daughters of  $^{241}\text{Pu}$ ,  $^{238}\text{U}$ , and  $^{232}\text{U}$  grow in. In gamma ray applications, the problem is manifested in the form of additional gamma rays which must be separated from the desired radiations. In calorimetry, the daughters contribute additional heat.

### 2.4.2 Interference to Stimulating Radiation

Material lowers the energy of neutrons traversing a container giving rise to an increase in the probability of inducing fissions. This problem becomes increasingly pronounced with low-atomic-number materials. Hydrogenous materials (e.g., water, plastics) have the strongest capability to produce this effect.

### 2.4.3 Attenuation of the Emitted Radiation

This effect may include the partial or complete loss of the energy of the emitted radiation. The detection of a reduced-energy radiation may mean that the radiation cannot be correctly assigned to its source. This effect can be severe for gamma ray systems. The effect increases with atomic number and the material density within the container. Also, systems which detect neutrons above a given energy will observe fewer neutrons above the given energy when low-atomic-number material is added to the container and thus produce a low assay indication.

The attenuation of the emitted radiation may be complete, as in the case of the absorption of neutrons in the nuclei of extraneous material. The probability for this absorption generally increases as the energy of the incident neutrons decreases. Hence, this effect is further aggravated when low-atomic-number materials are present to reduce the energy of the emitted neutrons.

### 2.4.4 Attenuation of the Stimulating Radiation

This phenomenon is similar to that of the preceding section. In this instance, the stimulating radiation does not penetrate to the SNM within the container and thus does not have the opportunity to induce fission. The presence of neutron poisons (e.g., Li, B, Cd, Gd) may attenuate the stimulating radiation to the extent that the response is independent of the SNM fissile content. Most materials absorb neutrons. The severity of this absorption effect is dependent on the type of material, its distribution, and the energy of the stimulating neutrons.

*The presence of extraneous material can thus alter the observed response, providing either a high or a low SNM content indication. This effect is further aggravated by nonuniformity within the container of either the*



*SNM or the matrix in which it is contained. This dependence is severe. Failure to attend to its ramifications through the segregation of scrap and waste categories and the utilization of representative calibration standards may produce gross inaccuracies in NDA measurements.*

## 2.5 Response Dependence on Container Dimensions and Composition

The items identified as potential sources of uncertainty in the observed response of an NDA system in Sections 2.1, 2.3, and 2.4 above can be minimized or aggravated through the selection of containers to be employed when assaying SNM contained in scrap or waste.

### 2.5.1 Container Dimensions

The practical limitation on container size for scrap and waste to be nondestructively assayed represents a compromise of throughput requirements and the increasing uncertainties in the observed NDA response incurred as a penalty for assaying large containers. Radiations emitted deep within the container must travel a greater distance to escape the confines of the container. Therefore, with increasing container size, the probability that radiations emitted near the center of the container will escape the container to the detectors decreases with respect to the radiations emitted near the surface of the container.

In active NDA systems, a relatively uniform field of stimulating radiation must be provided throughout that volume of the container which is observed by the detection system. This criterion is required to obtain a uniform response from a lump of SNM positioned anywhere within a container. It becomes increasingly difficult to satisfy this criterion and maintain a compact, geometrically efficient system with increasing container size. For this reason, the assay of small-size containers is recommended.

To facilitate loading into larger containers for storage or offsite shipment following assay, the size and shape of the inner and outer containers should be chosen to be compatible.

Packaging in small containers will produce more containers to be assayed for the same scrap and waste generation rates. An offsetting benefit, however, is that the assay accuracy of an individual container should be significantly improved over that of large containers. In addition, the total scrap and waste assay uncertainty should be reduced through statistically propagating a larger number of random component uncertainties to determine the total uncertainty.

### 2.5.2 Container Structural Composition

The structural composition of containers will affect

the penetration of the incident or the emerging radiation. Provided all containers are uniform, their effect on the observed response can be factored into the calibration of the system. The attainable assay accuracy will be reduced when containers with poor penetration or varying composition or dimensions are selected.

## 3. Nondestructive Assay for the Accountability for SNM Contained in Scrap and Waste

### 3.1 NDA Performance Objectives

The measurement accuracy objectives for any inventory component can be estimated by considering the amount of material typically contained in that inventory category. The measurement performance required is such that, when the uncertainty corresponding to the scrap and waste inventory component is combined with the uncertainties corresponding to the other inventory components, the quality constraints on the total limit of error of the material unaccounted for (LEMUF) will be satisfied.

### 3.2 NDA Technique Selection

NDA technique selection should reflect a consideration of the accuracy requirements for the assay and the type and range of scrap and waste categories to be encountered. No single technique appears capable of meeting all requirements. When more than one type of information is required to separate a composite response, more than one NDA technique may be required to provide that information.

#### 3.2.1 Plutonium Applications

Calorimetry determinations are the least sensitive to matrix effects, but rely on a detailed knowledge of the  $^{241}\text{Am}$  content and the plutonium isotopic composition to transform the measured heat flux to grams of plutonium.<sup>1</sup>

Gamma ray spectroscopy systems complement the potential of other assay methods by providing the capability to nondestructively determine, or verify, the  $^{241}\text{Am}$  content and the plutonium isotopic composition (except  $^{242}\text{Pu}$ ). High-resolution gamma ray systems are capable of extracting the maximum amount of information (isotopic composition, isotopic content, presence of extraneous gamma ray sources) from an assay, but content density severely affects the accuracy of quantitative predictions based upon that assay method.

Passive coincidence detection of the spontaneous fission yield of Pu-bearing systems provides an indication of the combined  $^{238}\text{Pu}$ ,  $^{240}\text{Pu}$ , and  $^{242}\text{Pu}$  sample content. With known isotopic composition, the Pu content can be computed.<sup>6</sup> Neutron multiplication effects become severe at high Pu sample loadings.<sup>7</sup>

Plastic scintillation coincidence detection systems are often designed in conjunction with active neutron interrogation source systems. Operated in passive and active modes, such systems are able to provide an assay of both the spontaneously fissioning and the fissile content of the sample. The spontaneous background can be subtracted from an active NDA response to provide a yield attributable to the fissile SNM content of the container.

Active NDA can be considered for plutonium scrap and waste applications after the potential implementation of the passive techniques has been evaluated. With the wide range of isotopic compositions encountered, together with the mixture with various enrichments of uranium, the requirements to convert an observed composite response into an accurate assay of the plutonium and uranium fissile content become increasingly severe.

The application of these methods to the assay of plutonium-bearing solids and solutions are the subjects of other Regulatory Guides.

### 3.2.2 Uranium Applications

Active neutron systems can provide for both high-energy and moderated interrogation spectrum capabilities. Operation with the high-energy neutron source will decrease the density dependence and neutron self-shielding effects, significantly enhancing the uniqueness of the observed response. To extend the applicability of such a system to small fissile loadings, a well-moderated interrogating spectrum can be used to take advantage of the increased  $^{235}\text{U}$  fission probability for neutrons of low energy. In highly enriched uranium scrap and waste (>20%  $^{235}\text{U}$ ), active NDA featuring a high-energy stimulating neutron flux is recommended.

The number and energy of the gamma rays emitted from the uranium isotopes (with the exceptions of the minor isotopes  $^{232}\text{U}$  and  $^{237}\text{U}$ ) are generally lower than for the plutonium case. The 185-keV transition observed in the decay of  $^{235}\text{U}$  is frequently employed in uranium applications. The penetration of this  $^{235}\text{U}$  primary gamma ray is so poor that the gamma ray NDA technique is not applicable with high-density, nonhomogeneous matrices.

There arise occasions when a passive enrichment determination is practical through the measurement of the 185-keV gamma ray. One criterion required for this application is that the contents be relatively homogeneous. This information can then be combined with an assay of the  $^{238}\text{U}$  content of the sample to compute the total uranium and  $^{235}\text{U}$  sample content. The  $^{238}\text{U}$  sample content can be obtained either through the detection of the  $^{238}\text{U}$  spontaneous fission neutron yield or through the assay of the  $^{234}\text{Pa}$  daughter gamma activity, provided either the  $^{234}\text{Pa}$  is in

equilibrium or its content is known. Enrichment meter applications for uranium will be the subject of another Regulatory Guide.

Calorimetry is not applicable to the assay of uranium enriched in the  $^{235}\text{U}$  isotope because of the low specific  $\alpha$  activity. In  $^{233}\text{U}$  applications, the intense activity of the daughter products of  $^{232}\text{U}$  imposes a severe complication on the use of calorimetry.

### 3.3 Categorization and Segregation of Scrap and Waste for NDA

The range of variations in the observed response of an NDA system attributable to the effects noted in Sections 2.3 and 2.4 above can be reduced or controlled. Following an analysis of the types of scrap and waste generated in conjunction with SNM processing, a plan to segregate scrap and waste at the generation points can be formulated. Recovery or disposal compatibility is important in determining the limits of each category. Limiting the range in variability in those extraneous NDA interference parameters discussed in Sections 2.3 and 2.4 is a primary means of improving the accuracy of the scrap and waste assay. Once the categories are established, it is important that steps be taken to assure that segregation into separate, uniquely identified containers occurs at the generation point.

Category limits can be established on the basis of measured variations observed in the NDA response of container loaded with a known amount of SNM. The variation in extraneous parameters can then be mocked up and the resultant effect measured. In establishing categories, the following specific items are significant sources of error.

#### 3.3.1 Calorimetry

The presence of extraneous materials capable of absorbing (endothermic) heat or emitting (exothermic) heat will cause the observed response to be less or greater than the correct response for the Pu in the sample.

#### 3.3.2 Neutron Measurements

The presence of high-yield ( $\alpha, n$ ) target material will increase the number of neutrons present in the sample. A fraction of these neutrons will induce fission in the fissile SNM isotopes and add another error to the measurement.

#### 3.3.3 Gamma Ray Measurements

Gamma rays are severely attenuated in interactions with heavy materials. Mixing contaminated combustibles with heavy, dense materials complicates the attenuation problem. Mixing of isotopic batches or mixing with radioactive non-SNM can also add to the complexity of the response.

### 3.3.4 Fission Measurements

Scrap or waste having low-atomic-number materials will reduce the energy of the neutrons present in the container, significantly affecting the probability of stimulating fission reactions.

Neutron-absorbing materials present in SNM scrap or waste may significantly affect the operation of NDA systems. Table B-1 of this guide identifies neutron absorbers in the order of decreasing probability of absorption of thermal neutrons. An estimate of the significance of the presence of one of these materials may be obtained from the ratio of its absorption cross section to the absorption cross section of the SNM present in the container:

$$R = \frac{N_1 \sigma_{a_1}}{N_{SNM} \sigma_{a_{SNM}}}$$

where

$N_1$  = the number of atoms per cubic centimeter of material,

$\sigma_{a_1}$  = absorption cross section of the extraneous material (Table B-1),

$N_{SNM}$  = number of atoms of SNM present per cubic centimeter,

$\sigma_{a_{SNM}}$  = absorption cross section of the SNM.

$$^{233}\text{U } \sigma_a = 573 \text{ barns}$$

$$^{235}\text{U } \sigma_a = 678 \text{ barns}$$

$$^{239}\text{Pu } \sigma_a = 1015 \text{ barns}$$

$$^{241}\text{Pu } \sigma_a = 1375 \text{ barns}$$

(Thermal neutron values)

TABLE B-1

### NATURALLY OCCURRING NEUTRON ABSORBERS<sup>8</sup>

Naturally Occurring Element	Symbol	Absorption Cross Section (barns)*	Naturally Occurring Element	Symbol	Absorption Cross Section (barns)*
Gadolinium	Gd	46,000	Terbium	Tb	46
Samarium	Sm	5,600	Cobalt	Co	38
Europium	Eu	4,300	Ytterbium	Yb	37
Cadmium	Cd	2,450	Chlorine	Cl	34
Dysprosium	Dy	950	Cesium	Cs	28
Boron	B	755	Scandium	Sc	24
Actinium	Ac	510	Tantalum	Ta	21
Iridium	Ir	440	Radium	Ra	20
Mercury	Hg	380	Tungsten	W	19
Protactinium	Pa	200	Osmium	Os	15
Indium	In	191	Manganese	Mn	13
Erbium	Er	173	Selenium	Se	12
Rhodium	Rh	149	Promethium	Pm	11
Thulium	Tm	127	Lanthanum	La	9
Lutetium	Lu	112	Thorium	Th	8
Hafnium	Hf	105	Iodine	I	7
Rhenium	Re	86	Antimony	Sb	6
Lithium	Li	71	Vanadium	V	5
Holmium	Ho	65	Tellurium	Te	5
Neodymium	Nd	46	Nickel	Ni	5

\*Cross section for thermal neutrons

The magnitude of this effect is dependent on the distribution of the materials and the energy of the neutrons present within the container. The relationship above is a gross approximation, and for convenience in calculation, including only the primary fissile isotope is sufficient to determine which materials may constitute a problem requiring separate categorization for assay. In extreme cases, either methods should be sought to measure the content of the neutron absorber to provide a correction for the NDA response or a different method should be sought for the assay of that category.

### 3.4 Packaging for Nondestructive Assay

Nondestructive assay provides optimal accuracy potential when the packages to be assayed are essentially identical and when the calibration standards represent those packages in content and form. Containers for most scrap and waste can be loaded using procedures which will enhance the uniformity of the loading within each container and from container to container. Compaction and vibration are two means to accomplish this objective.

### 3.5 Calibration of NDA Systems for Scrap and Waste

To obtain an assay value on SNM in a container of scrap or waste with an associated limit of error, the observed NDA response or the predicted content must be corrected for background and for significant effects attributable to the factors described in the preceding parts of this discussion.

The calibration of radiometric nondestructive assay systems is the subject of another Regulatory Guide.\*

One procedure for referencing NDA results to primary standards is the periodic selection of a container at random from a lot submitted for assay. That container should then be assayed a sufficient number of times to reduce the random uncertainty of the measurement to a negligible value. The SNM content of that container can then be determined through a different technique having an accuracy sufficient to verify the stated performance of the NDA system. This reference method should be traceable to primary standards. High-integrity recovery of the contents, followed by sampling and chemical analysis is one recommended technique.

## C. REGULATORY POSITION

In the development of an acceptable framework for the incorporation of nondestructive assay for the measurement of SNM-bearing scrap and waste, strong consideration should be given to technique selection,

\*To be based on ANSI N15.20, which is currently in development.

calibration, and operational procedures; to the segregation of scrap and waste categories; and to the selection and packaging of containers. The guidelines presented below are generally acceptable to the Regulatory staff for use in developing such a framework that can serve to improve materials accountability.

## 1. Analysis of Scrap and Waste

The origin of scrap and waste generated in conjunction with SNM processing activities should be determined as follows:

- a. Identify those operations which generate SNM-bearing scrap or waste as a normal adjunct of a process.
- b. Identify those operations which occasionally generate SNM-bearing scrap or waste as the result of an abnormal operation which renders the product unacceptable for further processing or utilization without treatment.
- c. Identify those scrap and waste items generated in conjunction with equipment cleanup, maintenance, or replacement.

The quantities of scrap and waste generated during normal operations in each category in terms of the total volume and SNM content should be estimated. Bulk measurement throughput requirements should be determined to assure that such assay will not constitute an operational bottleneck.

## 2. NDA Selection

### 2.1 Technique

The performance objectives for the NDA system should be derived as discussed in Section B.3.1. Techniques should be considered for implementation in the order of precedence established in Table C-1 of this guide. Selection should be based on attainable accuracy, factoring into consideration the characteristics of the scrap and waste categories. The application of such techniques will be the subjects of other Regulatory Guides.

### 2.2 System Specifications

NDA systems for SNM accountability should be designed and shielding should be provided to meet the following objectives:

- a. Performance characteristics should be essentially independent of fluctuations in the ambient operational environment, including:
  - (1) External background radiations,
  - (2) Temperature,
  - (3) Humidity, and
  - (4) Electric power.
- b. Response should be essentially independent of positioning of SNM within the scrap or waste container including effects attributable to:

**TABLE C-1  
NDA TECHNIQUE SELECTION**

TECHNIQUE	Pu	<sup>235</sup> U	>20% <sup>235</sup> U	<20% <sup>235</sup> U
(1) CALORIMETRY	1st (1+2)*	3rd	NA	NA
	NR	NR	NA	NA
(2) GAMMA RAY	3rd	2nd	2nd	1st (2+5)
	1st	1st	1st	1st
(3) SPONTANEOUS FISSION	2nd (3+2)	NA	NR	3rd (3+2)**
	2nd (3+2)	NA	NR	NR
(4) STIMULATED FISSION	4th	1st	1st	2nd
	3rd	2nd	2nd	2nd
(5) GROSS NEUTRON	NR	NR	NIR (5+2)	NIR (5+2)
	NR	NR	NR	NR

\*Above recommendation refers to high-density, high-SNM-content items. Lower recommendation refers to low-density, low-SNM items.

\*\*Spontaneous fission of <sup>235</sup>U.

NR-NOT RECOMMENDED-Technique marginal for this application.

NA-NOT APPLICABLE.

NIR-NOT INDEPENDENTLY RECOMMENDED-Should be used only in conjunction with a complementary assay method.

**TABLE C-2  
NDA INTERFERENCE CONTROL**

NDA Technique	Heat Producing or Absorbing Processes	Mixed SNM	Mixed Isotopic Batches	Miscellaneous Radiations	Presence of High-Yield (α,n) Target Material	Gamma Ray Absorbers	Neutron Absorbers	Neutron Moderating Materials	Lumped vs. Distributed SNM	Lumped vs. Distributed Matrix Mat'l
Calorimetry	xxx	—	xxx			—	—	—	—	—
Gamma Ray Spectroscopy	—	x	x	x	—	xxx	—	—	xxx	xx
Spontaneous Fission Detection	—	xx	xxx	xx <sup>a</sup>	xx <sup>b</sup>	xx <sup>a</sup>	xx <sup>c</sup>	xx	xx	x
Stimulated Fission Detection	—	xxx	xxx	xx <sup>a</sup>	xx <sup>b</sup>	xx <sup>a</sup>	xxx <sup>c</sup>	xx <sup>d</sup> xxx <sup>e</sup>	xxx <sup>d</sup> x <sup>e</sup>	x <sup>a</sup>

**Key:**  
 — No apparent sensitivity.  
 x Some sensitivity. Evaluate effect in extreme cases.  
 xx Marked sensitivity. Categorize and calibrate according to magnitude of observed effect.  
 xxx Strong sensitivity. Requires correction to assay. May render technique unacceptable in extreme cases if correction not possible

**Notes:** a — Effect depends on type and nature of radiation detected.  
 b — Effect less pronounced in coincidence detection systems.  
 c — Same as a, additional effect due to neutron multiplication.  
 d — Moderated-neutron stimulating source.  
 e — High-energy stimulating source.

- (1) Detector geometrical efficiency, and
- (2) Stimulating source intensity and energy.

Techniques to achieve these objectives are discussed in Section B of this guide.

### 3. Categorization

Scrap and waste categories should be developed on the basis of NDA interference control, recovery or disposal compatibility,<sup>9</sup> and relevant safety considerations. Categorization for NDA interference control should be directed to limiting the range of variability in an interference. Items to be considered depend upon the sensitivity of the specific NDA technique, as shown in Table C-2.

The means through which these interferences are manifested are detailed in Section B. When such effects or contents are noted, separate categories should be established wherein the materials are isolated.

### 4. Containers

#### 4.1 Size Constraints

Scrap and waste should be packaged for assay in containers as small as practicable, consistent with the capability and sensitivity of the NDA system.

To enhance the penetration of stimulating or emitted radiations containers should be cylindrical. The diameter should be less than five inches to provide for significant loading capability, ease in loading, reasonable penetrability characteristics, and compatibility with criticality-safe geometry requirements for individual containers, where applicable.

Containers having an outside diameter of 4-3/8 inches will permit nineteen such containers to be arranged in a cross section of a 55-gallon drum, even when that drum contains a plastic liner. Containers having an overall length equal to some integral fraction of the length of a 55-gallon drum are further recommended when shipment or storage within such containers is to be considered. For normal operations, an overall length of either 16-1/2 inches (two layers or 38 containers per drum) or 11 inches (three layers or 57 containers per drum) is therefore recommended.

#### 4.2 Structural Features

Containers should be selected in accordance with normal safety considerations and should be:

- a. Structurally identical for all samples to be assayed within each category,
- b. Structurally identical for as many categories as practicable to facilitate loading into larger containers or storage facilities,
- c. Uniform in wall thickness and material composition,
- d. Fabricated of materials that do not significantly

interfere with the radiations entering or leaving the sample,

- e. Capable of being sealed to verify post-assay integrity, and
- f. Compatible with subsequent recovery, storage, and disposal requirements, as applicable.

In most NDA applications, uniformity of composition is more important than the specification of a particular material. Table C-3 gives general recommendations for container structural materials.

TABLE C-3

### SCRAP AND WASTE CONTAINER COMPOSITION

NDA Technique	Container Composition
Calorimetry	metal (aluminum, brass)
Gamma Ray Analysis	cardboard, polyethylene bottle, thin metal
Spontaneous or Stimulated Fission	thin metal, cardboard, polyethylene bottle
Gross Neutron	thin metal, cardboard, polyethylene bottle

#### 4.3 Container Identification

To facilitate loading and assay within the segregation categories, containers should either be uniquely color-coded or carry unique color-coded identification labels. Identification of categories should be documented and operating personnel instructed to assure compliance with established segregation objectives.

### 5. Packaging

Containers, where practical, should be packaged with a quantity of material containing sufficient SNM to assure that the measurement is not being made at the extremes of the performance bounds for that system. Packaging procedures should be consistent with relevant safety practices.

Containers should be packaged in as reproducible a manner as possible. Low-density items should be compacted to reduce bulk volume and to increase the container SNM loading. Lowering the bulk volume reduces the number of containers to be assayed and generally improves the assay precision.

If assay predictions are significantly affected by the variability in the distribution of the container contents, compacting or vibrating the container on a shake table to settle the contents should be used to enhance the assay accuracy in conjunction with rotating and scanning the container during assay.

#### 6. Calibration

The NDA system(s) should be independently calibrated for each category of scrap or waste to be assayed.

Within each category, the variation of interference effects should be measured within the boundaries defining the limits of that category. Calibration standards should employ containers identical to those to be employed for the scrap or waste. Their contents should be mocked up to represent the range of variations in the interferences to be encountered. To minimize the number of standards required, the calibration standards should permit the range of interference variations to be simulated over a range of SNM loadings.

Calibration relationships should be verified at intervals sufficiently frequent to detect deviations from the expected response in time to make corrections before the containers are processed or shipped.

Assay values should be periodically verified through an independent measurement using a technique sufficiently accurate to resolve NDA uncertainty. Periodically, a container of scrap or waste should be randomly selected for verification. Once selected, the NDA analysis should be repeated a minimum number of five times to determine the precision characteristics of

the system. The contents of that container should then be independently measured using one of the following techniques:

- a. Recovery of the contents, followed by sampling and chemical analysis.
- b. High-accuracy calorimetry (Pu only) with isotopic sample taken from contents and determined through standard techniques.
- c. Small-sample screening followed by selective chemical analyses. This technique is applicable to cases in which the contents consist of a collection of similar items. Each item should be assayed in a small-sample system capable of an accuracy greater than or equal to that of the system being calibrated. No less than five items should then be selected for chemical analysis. Those items should be chosen to span the range of observed responses in the screening assay.

Verification measurements should be used to periodically update calibration data when the comparison with predicted quantities is satisfactory. Calibration of the system is not acceptable when the NDA predicted value does not agree with the measured value to within the value of the combined limits of error:

$$|NDA-VER| \leq (LE_{NDA}^2 + LE_{VER}^2)^{1/2}$$

Calibration data and hypotheses should be reinvestigated when this criterion is not satisfied.

The calibration of NDA systems will be the subject of another Regulatory Guide.

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