



# REGULATORY GUIDE

## OFFICE OF NUCLEAR REGULATORY RESEARCH

### REGULATORY GUIDE 6.21 (Task SG 044-4)

## NONDESTRUCTIVE URANIUM-235 ENRICHMENT ASSAY BY GAMMA RAY SPECTROMETRY

### A. INTRODUCTION

Section 70.51, "Material Balance, Inventory, and Records Requirements," of 10 CFR Part 70, "Domestic Licensing of Special Nuclear Material," requires, in part, that licensees authorized to possess and use at any one time more than one effective kilogram of special nuclear material (SNM) determine the inventory difference (ID) and its associated standard error (estimator) of inventory difference (SEID) for each element and the fissile isotope for uranium contained in material in process. Such a determination is to be based on measurements of the quantity of the element and of the fissile isotope for uranium.

The majority of measurement techniques used in SNM accountability are specific to either the element or the isotope but not to both. A combination of techniques is therefore required to determine the ID and SEID by element and by fissile isotope for uranium. Passive gamma ray spectrometry is a nondestructive method for measuring the enrichment or relative concentration of the fissile isotope  $^{235}\text{U}$  in uranium, but this technique is used in conjunction with an assay for the element uranium in order to determine the amount of  $^{235}\text{U}$ .

This guide describes conditions for  $^{235}\text{U}$  enrichment measurements using gamma ray spectrometry that are acceptable to the NRC staff and provides procedures for operation, calibration, error analysis, and measurement control.<sup>1</sup> Examples of  $^{235}\text{U}$  enrichment assays using portable and in-line instruments based on the techniques outlined in this guide may be found in References 1 through 4.

Any guidance in this document related to information collection activities has been cleared under OMB Clearance No. 3150-0009.

<sup>1</sup> Calibration, error analysis, and measurement control are discussed in Regulatory Guide 5.53, "Qualification, Calibration, and Error Estimation Methods for Nondestructive Assay." A proposed revision to this guide has been issued for comment as Task SG 049-4.

### B. DISCUSSION

#### 1. BASIS FOR GAMMA RAY MEASUREMENT OF URANIUM ENRICHMENT

The alpha decay of  $^{235}\text{U}$  to  $^{231}\text{Th}$  is accompanied by the emission of a prominent gamma ray at 185.7 keV ( $4.3 \times 10^4$  of these 185.7-keV gamma rays are emitted per second per gram of  $^{235}\text{U}$ ). The relatively low energy and consequent low penetrating power of these gamma rays implies that most of the rays that are emitted in the interior of the sample are absorbed within the material itself. Thick<sup>2</sup> materials therefore exhibit a 185.7-keV gamma ray emission characteristic of an infinite medium; i.e., the 185.7-keV gamma flux emitted from the sample surface does not depend upon the size or dimensions of the material. Under these conditions, the 185.7-keV intensity is directly proportional to the  $^{235}\text{U}$  enrichment. A measure of this 185.7-keV intensity with a suitable detector forms the basis for an enrichment measurement technique.

The thickness of the material with respect to the mean free path of the 185.7-keV gamma ray is the primary characteristic that determines the applicability of passive gamma ray spectrometry for the measurement of isotope enrichment. The measurement technique is applicable only if the material is thick. However, in addition to the thickness of the material, other conditions must be satisfied before the gamma ray measurement technique can be accurately applied. An approximate analytical expression for the detected 185.7-keV activity is given below. This expression has been separated into several individual terms to aid in identifying those parameters that may interfere with the measurement. Although approximate, this relationship can be used to estimate the magnitude of

<sup>2</sup> The terms "thick" and "thin" are used throughout this guide to refer to distances in relation to the mean free path of the 185.7-keV gamma ray in the material under consideration. The mean free path is the 1/e-folding distance of the gamma ray flux or, in other terms, the average distance a gamma ray traverses before interacting.

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interfering effects in order to establish limits on the range of applicability and to determine the associated uncertainties introduced into the measurement. This relationship is:

$$C = E (a/\mu_U) A \left[ 1 + \sum_i \frac{\rho_i \mu_i}{\rho_U \mu_U} \right]^{-1} \epsilon (\Omega/4\pi) \exp(-\rho_c \mu_c d) \quad (1)$$

effective source of 185.7-keV gamma rays seen by the detector

where

C = detected 185.7-keV activity

E = enrichment of the uranium (<1)

$\rho_U, \rho_i, \rho_c$  = density of the uranium (U), matrix material (i), and container wall (c), respectively, in g/cm<sup>3</sup>

$\mu_U, \mu_i, \mu_c$  = mass attenuation coefficient for 185.7-keV gamma rays in uranium (U), matrix material (i), and container wall (c) in cm<sup>2</sup>/g

a = specific 185.7-keV gamma ray activity of <sup>235</sup>U  
= 4.3 x 10<sup>4</sup> gamma rays/sec-g

$\epsilon$  = net absolute detector full energy peak efficiency for detecting 185.7-keV gamma rays ( $\epsilon \leq 1$ )

$\Omega$  = solid angle subtended by the detector ( $\Omega \leq 2\pi$ )

A = cross-sectional area of material defined by the detector collimator

d = container wall thickness

A derivation of this expression, as well as other necessary background information on the theory of enrichment measurements, may be found in Reference 5. As evident in Equation 1, the activity (C) is proportional to the enrichment (E) but is affected by several other characteristics as well.

## 2. MATERIAL AND CONTAINER WALL EFFECTS ON MEASUREMENT

### 2.1 Material Thickness

In order for Equation 1 to be applicable, the material must be sufficiently thick to produce strong attenuation of 185.7-keV gamma rays. To determine whether this criterion is met, it is useful to compare the actual thickness of the

material with a characteristic length called the critical distance  $x_0$ , where  $x_0$  is defined as the thickness of material that produces 99.5 percent of the measured 185.7-keV activity:

$$x_0 = -\lambda \ln(0.005) = 5.29\lambda \quad (2)$$

where

$$1/\lambda = \mu_U \rho_U + \sum_i \mu_i \rho_i \quad (3)$$

Calculated values of  $x_0$  for several common materials are given in Table 1.

Table 1\*

### CALCULATED VALUES OF $x_0$ AND MATERIAL COMPOSITION TERM

Material	Density (g/cm <sup>3</sup> )	Critical Distance $x_0$ (cm)	Material Composition Term $1 + \sum_i \frac{\rho_i \mu_i}{\rho_U \mu_U}$
U (metal)	18.7	0.20	1.000
UF <sub>6</sub>	4.7	1.08	1.040
UO <sub>2</sub>	10.9	0.37	1.012
U <sub>3</sub> O <sub>8</sub>	7.3	0.56	1.015
Uranyl Nitrate	2.8	2.30	1.095

\* Values of the mass attenuation coefficient,  $\mu$ , may be found in References 6 and 7.

Other nondestructive assay (NDA) techniques are capable of detecting SNM distributed within a container. The enrichment measurement technique, however, is inherently a surface measurement. Therefore, the "sample" observed, i.e., the surface, must be representative of all the material in the container. In this respect the enrichment measurement is more analogous to chemical analysis than are other NDA techniques.

### 2.2 Material Composition

If the gamma ray measurement is to be dependent only on the enrichment, the term related to the composition of the matrix should be approximately equal to one, i.e.,

$$1 + \sum_i \frac{\rho_i \mu_i}{\rho_U \mu_U} \approx 1 \quad (4)$$

This condition ensures that the enrichment measurement will be insensitive to variations in the matrix composition. However, if this matrix term differs significantly from unity, the enrichment measurement can still be performed provided the matrix composition of the standard and samples remains reasonably constant.

Calculated values of this quantity for common materials are given in Table 1. The deviations of the numbers in Table 1 from unity indicate that a bias can be introduced by ignoring the difference in material composition.

Inhomogeneities in matrix material composition, uranium density, and uranium enrichment within the measured volume of the material (as characterized by the depth  $x_0$  and the collimated area A) can produce changes in the measured 185.7-keV activity and affect the accuracy of an enrichment calculated on the basis of that activity. Variations in the content of low-atomic-number ( $Z < 30$ ) matrix materials and inhomogeneities in uranium density in such matrix material produce a small to negligible effect on measurement accuracy. Care is necessary, however, in applying this technique to materials having high-atomic-number matrices ( $Z > 50$ ) or materials having uranium concentrations less than approximately 75 percent. Significant inaccuracies can arise when the uranium enrichment itself varies throughout the sample.

The above conclusions about the effects of inhomogeneities are based on the assumption that the thickness of the material exceeds the critical distance,  $x_0$ , and that the inhomogeneities exist within this depth. In the case of extremely inhomogeneous materials such as scrap, the condition of sufficient depth may not always be fulfilled or inhomogeneities may exist beyond the depth  $x_0$ ; i.e., the sample is not representative. Therefore, this technique is not applicable to such inhomogeneous materials.

### 2.3 Container Wall Thickness

Variations in the thickness of the container walls can significantly affect the activity measured by the detector. The fractional change in the activity  $\Delta C/C$  due to a small change  $\Delta d$  in the container wall thickness can be expressed:

$$\frac{\Delta C}{C} = -\mu_c \rho_c \Delta d \quad (5)$$

Calculated values of  $\Delta C/C$  corresponding to a change in container thickness  $\Delta d$  of 0.0025 cm for common container materials are given in Table 2.

Table 2

#### CALCULATED VALUES OF $\Delta C/C$

Material	Density (g/cm <sup>3</sup> )	$\frac{\Delta C}{C}$
Steel	7.8	-0.003
Aluminum	2.7	-0.0009
Polyethylene	0.95	-0.0004

Therefore, the container wall thickness must be known (e.g., by measuring an adequate number of the containers before loading). In some cases, an unknown container wall thickness can be measured using an ultrasonic technique after which a simple correction can be applied to the data

to account for attenuation of the 185.7-keV gamma rays (see Equation 5). Commercial equipment is available to measure wall thicknesses ranging from about 0.025 to 5.0 cm with relative precisions of approximately 1.0 percent to 0.1 percent, respectively.

Using standardized containers to hold the sample material in order to minimize uncertainties and possible errors associated with container-to-container wall thickness corrections is strongly recommended.

## 3. DETECTOR-RELATED FACTORS

### 3.1 Area and Geometrical Efficiency

The area of the material viewed by the detector and the geometrical efficiency are variables that may be adjusted, within limits, to optimize a system. Two important factors must be noted:

1. Once these variables are fixed, changes in these parameters will alter the calibration of the instrument and invalidate subsequent measurement results.

2. The placement of the material within the container will affect the detected activity. It is important that there are no void spaces between the material and the container wall.

### 3.2 Net Detector Efficiency

Thallium-activated sodium iodide, NaI(Tl), lithium-drifted germanium, Ge(Li), and high-purity germanium, HPGe (also referred to as intrinsic germanium, IG), detectors have been used to perform these measurements. The detection systems are generally conventional gamma ray spectrometry systems that are commercially available in modular or single-unit construction. Some useful guidelines for the procurement and setup of a solid-state-detector-based system are given in Regulatory Guide 5.9, "Specifications for Ge(Li) Spectroscopy Systems for Material Protection Measurements."<sup>3</sup>

Factors that influence detector selection and the control required for accurate results are discussed below.

#### 3.2.1 Background

**3.2.1.1 Compton Background.** This background is predominantly produced by the 765-keV and 1001-keV gamma rays of <sup>234m</sup>Pa, a daughter of <sup>238</sup>U. Since in most cases the Compton background behaves smoothly in the vicinity of the 185.7-keV peak, it can be readily subtracted, leaving only the net counts in the 185.7-keV full-energy peak.

**3.2.1.2 Overlapping Peaks.** The observable peak from certain gamma rays may overlap that of the 185.7-keV peak owing to the finite energy resolution of the detector; i.e.,

<sup>3</sup>A proposed revision to this guide has been issued for comment as Task SG 042-2 with the title "Guidelines for Germanium Spectroscopy Systems for Measurement of Special Nuclear Material."

the difference in energies may be less than twice the full width of the spectrum peak at half its maximum height (FWHM). This problem is common in enrichment measurements of recently separated uranium from a reprocessing plant. The peak from a strong 208-keV gamma ray from  $^{237}\text{U}$  (half-life of 6.75 days) can overlap the 185.7-keV peak when a NaI detector is used. Analytical separation of the two unresolved peaks, i.e., peak stripping, may be applied. An alternative solution is to use a Ge(Li) or HPGe detector so that both peaks are clearly resolved. The  $^{237}\text{U}$  activity present in reprocessed uranium will depend on the amount of  $^{241}\text{Pu}$  present before reprocessing and also on the time elapsed since separation.

**3.2.1.3 Ambient Background.** The third source of background originates from natural sources and from other uranium-bearing materials located in the vicinity of the measuring apparatus. This source can be particularly bothersome since it can vary over time within wide limits depending on plant operating conditions.

### 3.2.2 Count-Rate Losses

Calculation of the detector count rates for purposes of making dead-time<sup>4</sup> estimates requires calculation of the total count rate, not only that due to  $^{235}\text{U}$ . Total count-rate estimates for low-enrichment material must therefore take into account the relatively important backgrounds of gamma rays from  $^{238}\text{U}$  daughters. If other radioactive materials are present within the sample, their contributions to the total count rate must also be considered.

Count-rate corrections can be made by determining the dead time or by making measurements for known live-time<sup>4</sup> intervals. The pileup or overlap of electronic pulses is a problem that also results in a loss of counts in the full-energy peak for Ge(Li) systems. An electronic pulser may be used to monitor and correct for these losses. However, a more reliable method involves the use of a radioactive source fixed to the detector in an invariant geometry. A photopeak area from the spectrum of this source is counted along with a uranium peak area. The source peak area can then be compared with an earlier value taken without uranium present, and the dead time for the assay measurement can be inferred. (Part of the regular measurement control would then involve uranium-free measurement of the source peak area.) One possible source could be  $^{241}\text{Am}$ , whose 60-keV gamma ray peak would be easily resolved from the uranium lines by either a Ge- or NaI-based system. If filtering of ambient low-energy gamma radiation is used, the  $^{241}\text{Am}$  source can be placed between the detector and the absorber used for the filtering. If a high-resolution system is used, the recommended source for this purpose is  $^{109}\text{Cd}$ , which emits only an 88-keV peak, well below the uranium (185.7-keV) region, and has a half-life of 453 days. Radiation that provides no useful information can be selectively attenuated by filters; e.g., a

<sup>4</sup>“Dead time” refers to that portion of the measurement period during which the instrument is busy processing data already received and cannot accept new data. “Live time” means that portion of the measurement period during which the instrument can record detected events. To compare different data for which dead times are appreciable, compare counts measured for equal live-time periods, i.e., (actual measurement period) - (dead time) = live time.

1-mm-thick cadmium filter will reduce x-ray interference, eliminating this source of count-rate losses. Note that present-day counting electronics are capable of handling high negative count rates without significant losses from either pileup or system dead time. However, if a measurement situation arises in which count rates are excessive, tighter collimation of the opening on the front face of the detector is a simple method for reducing count rates to tolerable levels at which complicated loss corrections are not essential.

### 3.2.3 Instability in Detector Electronics

The gain of a photomultiplier tube is sensitive to changes in temperature, count rate, and magnetic field. Provision can be made for gain checks or gain stabilization for enrichment measurement applications. Various gain stabilizers that automatically adjust the system gain to keep a reference peak centered between two preset energy limits are available.

## C. REGULATORY POSITION

Passive gamma ray spectrometry constitutes a means acceptable to the NRC staff for nondestructively determining  $^{235}\text{U}$  enrichment, if the conditions identified below are satisfied.

### 1. RANGE OF APPLICATION

All material to be assayed under a certain calibration should be of similar chemical form, physical form, homogeneity, and impurity level.

The critical distance of the material should be determined. Only those items of the material having dimensions greater than this critical distance should be assayed by this technique.

The material should be homogeneous in all respects on a macroscopic<sup>5</sup> scale. The material should be homogeneous with respect to uranium enrichment on a microscopic<sup>5</sup> scale.

The containers should all be of similar size, geometry, and physical and chemical composition.

### 2. SYSTEM REQUIREMENTS

NaI(Tl) scintillation detectors having a resolution of FWHM less than 16 percent at the 185.7-keV peak of  $^{235}\text{U}$  are generally adequate for measuring the enrichment of uranium. Crystals with a thickness in the range of 1.3 to 1.8 cm are recommended for optimum efficiency. If other radionuclides that emit significant quantities of gamma radiation in an energy region  $E = 185.7 \text{ keV} \pm 2 \text{ FWHM}$  at 185.7 keV are present, one of the following should be used:

- a. A higher resolution detector, e.g., Ge(Li) or HPGe, or

<sup>5</sup>Macroscopic refers to distances greater than the critical distance; microscopic to distances less than the critical distance.

b. A peak-stripping procedure to subtract the interference. In this case, data should be provided to show the range of concentration of the interfering radionuclide and the accuracy and precision of the stripping technique over this range.

The detection system gain should be stabilized by monitoring a known reference peak.

The system clock should be in live time. The system should provide a means of determining the count-rate losses based on the total counting rate, or provide additional collimation to reduce the count rate.

The design of the system should allow reproducible positioning of the detector or item being assayed.

The system should be capable of determining the gamma ray activity in at least two energy regions to allow subtraction of the background. One region should encompass 185.7 keV, and the other should be above this region but should not overlap it. The threshold and width of the regions should be adjustable. If dead-time corrections are measured with a pulser or source peak, a third and fourth region will have to be defined to establish the additional peak area and its background.

The system should have provision for filtering out low-energy radiation from external sources.

### 3. DATA ACQUISITION

Initial preparation of the assay instrumentation for data acquisition should involve careful determination of the system energy gain, the position of key photopeak and background regions, and the instrument response to calibration. However, after the proper instrument settings are established, routine operation can involve a less detailed check of the peak positions. This verification can consist of either a visual check of the gamma ray spectrum on a multichannel analyzer or a brief scan of the 140- to 200-keV energy region with a single-channel analyzer. Verification that the 185.7-keV peak position corresponds to its value at calibration ensures that the instrument is still biased properly. Verification of the 185.7-keV count rate with a uranium check source can also demonstrate continued validity of the response calibration. In some cases it may be useful to check the position of two peaks in the gamma ray spectrum, in which case a  $^{57}\text{Co}$  gamma ray source (with a photopeak at 122 keV) would be convenient.

If the total counting rate is determined primarily by the 185.7-keV gamma ray, the counting rate should be restricted (e.g., by absorbers or decreased geometrical efficiency) below those rates requiring correction. The system sensitivity will be reduced by these measures, and, if the sensitivity is no longer adequate, separate calibrations should be made in two or more enrichment regions.

To determine the location and width of the 185.7-keV peak region and the background regions, the energy spectrum from each calibration standard (see Regulatory Position 4,

Calibration) should be determined and the position of the 185.7-keV peak and neighboring peaks noted. The threshold and width of each energy region should then be selected to avoid including any neighboring peaks and to optimize the system stability and the signal-to-background ratio.

The net response attributed to 185.7-keV gamma rays should be the accumulated counts in the peak region minus a multiple of the counts accumulated in a nearby background region. A single upper background region may be monitored, or both a region above the peak region and one below may be monitored. If only an upper background region is monitored, the net response,  $R$ , is given by

$$R = G - bB$$

where  $G$  and  $B$  are the gross counts in the peak region and the background region, respectively, and  $b$  is the multiple of the background to be subtracted. This net response,  $R$ , should then be proportional to the enrichment,  $E$ :

$$E = C_1 R = C_1 (G - bB)$$

where  $C_1$  is a calibration constant to be determined (see Regulatory Position 4, Calibration). The gross counts,  $G$  and  $B$ , should be measured for all the standards. The quantities  $G/E$  should then be plotted as a function of the quantities  $B/E$  and a straight line through the data determined:

$$G/E = b(B/E) + 1/C_1$$

The slope of this line is  $b$ , the multiple of the upper background region to be subtracted. The data from all the standards should be used in determining this slope.

If both an upper and a lower background are monitored, the counts in each of these regions should be used to determine a straight-line fit to the background. Using this straight-line approximation, the area or number of counts under this line in the peak region should be subtracted from the gross counts,  $G$ , to obtain the net response. An adequate technique based on this principle is described in Reference 8. On a number of recently developed portable gamma ray spectroscopy instruments, these calibration procedures can be performed automatically by means of a microprocessor-based computational capability built into the instrument or by a calculator. In such cases, the more reliable procedure of complete calibration of the instrument before each assay session may be practical.

### 4. CALIBRATION

Calibration<sup>6</sup> standards should be obtained by:

1. Selecting items from the production material. A group of the items selected should, after determination of

<sup>6</sup>None of the calibration techniques or data reduction procedures discussed precludes the use of automated direct-readout systems for operation. The procedures described in this guide should be used for adjustment and calibration of direct-readout instruments.

the gamma ray response, be measured by an independent, more accurate technique, e.g., mass spectrometry, that is traceable to or calibrated with National Bureau of Standards (NBS) standard reference material. The other items should be retained as working standards.

2. Fabricating standards that represent the material to be assayed in chemical form, physical form, and impurity level. The  $^{235}\text{U}$  enrichment of the material used in the fabrication of the standards should be determined by a technique, e.g., mass spectrometry, that is traceable to or calibrated with NBS standard reference material.

The containers for the standards should have a geometry, dimensions, and a composition that approximate the mean of these parameters in the containers to be assayed. However, it should be emphasized that the best procedure is to standardize the sample containers to minimize, if not eliminate, container-to-container differences.

3. The values of enrichment for the calibration standards should span the range of values encountered in normal operation. No less than three separate standards should be used. (Good calibration practice dictates the use of at least two standards to determine the linear calibration constants and a third standard to check the calibration computations.) However, if the assay response (after application of appropriate corrections) can be shown to be highly linear and to have zero offset (i.e., zero response for zero enrichment), it may be more advantageous to avoid using standards with low enrichment because the low count rates would reduce the calibration precision. In such a case, calibration in the upper half of the range of expected enrichments combined with the constraint of zero response for zero enrichment can produce a higher precision calibration than a fitting of standard responses over the full range of expected enrichments, including values at low enrichment. If such a calibration procedure is used, careful initial establishment of the zero offset and instrument linearity, followed by occasional verification of both assumptions, is strongly recommended. Such verification could be accomplished by an occasional extended measurement of a low-enrichment standard. It should be noted that if the measurement system exhibits a nonzero offset (i.e., a nonzero response for zero sample enrichment), this is an indication of a background problem that should be corrected before assays are performed.

Each standard should be measured at a number of different locations, e.g., for a cylinder, at different heights and rotations about the axis. The mean of these values should be used as the response for that enrichment. The dispersion in these values should be used as an initial estimate of the variance due to material and container inhomogeneity.

In general, the data from the standards, i.e., the net responses attributed to the 185.7-keV gamma rays from the known uranium enrichments, can be employed in a simple linear calculation of the two calibration constants as described in Appendix 3 of Reference 5. If desired, more involved least-squares techniques can also be used.

## 5. OPERATIONS

The measurement of enrichment involves counting the 185.7-keV gamma ray intensity from an infinite thickness of uranium-bearing material in a constant counting geometry. A schematic of the counting geometry is given in Figure 1. The detector should be collimated and shielded from ambient radiation so that, as much as possible, only the radiation from the sample container is detected.

The detection system and counting geometry (i.e., collimator opening area,  $A$ , and collimator depth,  $x$ ), the data reduction technique, and the count-rate loss corrections, if included, should be identical to those used in the calibration.

Data from all measurements should be recorded in an appropriate log book.

At least two working standards should be measured during each eight-hour operating shift. The measured response should be compared to the expected response (value used in calibration) to determine if the difference exceeds three times the expected standard deviation.<sup>7</sup> If this threshold is exceeded, measurements should be repeated to verify that the response is significantly different and that the system should be recalibrated. In the event of a significant change in the instrument response, every effort should be made to understand the underlying cause of the change and, if possible, to remedy the cause rather than simply calibrate around the problem.

Prior to counting, all containers should be agitated. If this is not possible, the material should be mixed by some method. One container from every ten should be measured at two different locations on the container. The others may be measured at only one location. (If containers are scanned to obtain an average enrichment, the degree of inhomogeneity should still be measured by this method.)

The difference between the measurements at different locations on the container should be used to indicate a lack of the expected homogeneity. If the two responses differ by more than three times the expected standard deviation (which should include the effects of the usual or expected inhomogeneity), measurements should be repeated to verify the existence of an abnormal inhomogeneity. If the threshold is exceeded, the container should be rejected and investigated to determine the cause of the abnormal inhomogeneity.<sup>8</sup>

The container should be viewed at such a position that an infinite thickness of material fills the field of view defined by the collimator and detector (see Figure 1). The procedure for determining the fill of the container should be recorded, e.g., by visually inspecting at the time of filling and recording on the container tag.

<sup>7</sup>The user can always have a stricter criterion. This is a minimum.

<sup>8</sup>The difference may also be due to a large variation in wall thickness.

## SCHEMATIC OF ENRICHMENT MEASUREMENT SETUP

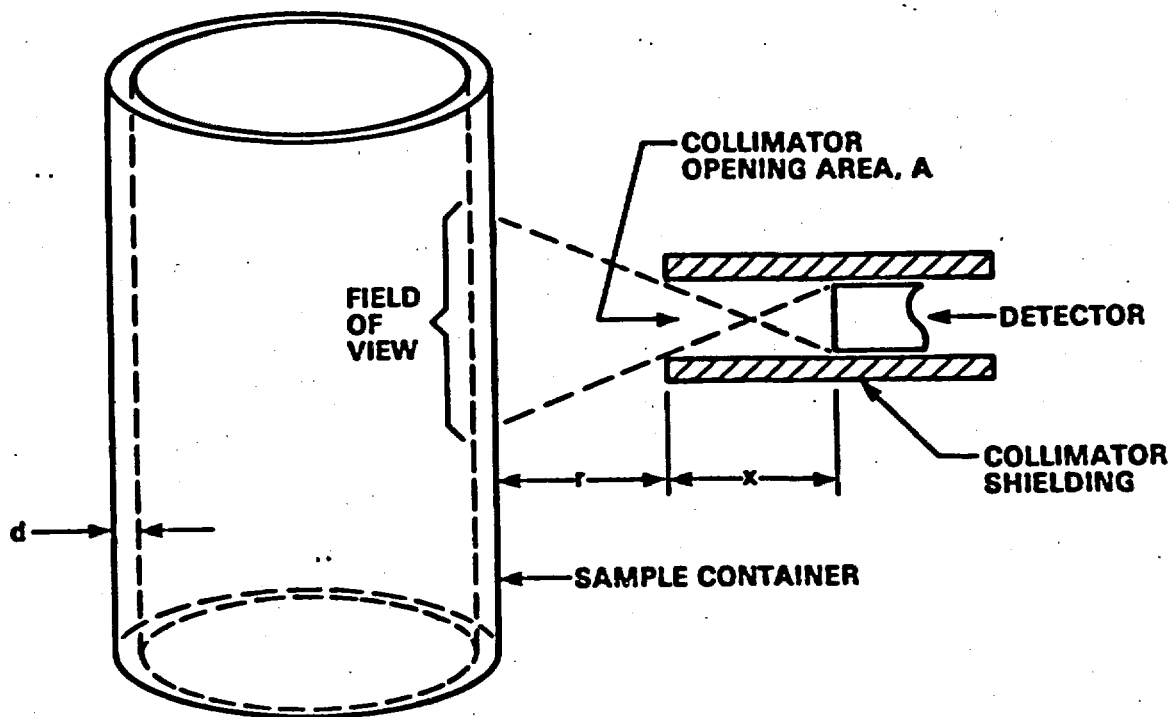


FIGURE 1

A schematic of a typical detector/collimator arrangement for a uranium enrichment measurement. The collimator depth (crucial in the calibration of the enrichment instrument) is denoted by  $x$ , the distance from the container surface to the collimator opening by  $r$ , and the container wall thickness by  $d$ . As long as an infinite thickness of assay material is contained in the field of view of the detector, the distance  $r$  is not crucial. However, the preferred enrichment measurement setup is with the collimator opening in contact with the container surface (i.e.,  $r = 0$ ).

The container wall thickness should be measured. The wall thickness and location of the measurement should be indicated if the individual wall thickness measurements and the gamma ray measurement are made at this location. If the containers are nominally identical, an adequate sampling of these containers should be sufficient. The mean of the measurements on these samples constitutes an acceptable measured value of the wall thickness that may be applied to all containers of this type or category.

The energy spectrum from a process item selected at random should be used to determine the existence of unexpected interfering radiations and the approximate magnitude of the interference. This test should be performed at a frequency that will ensure testing:

1. At least one item in any new batch of material.
2. At least one item if any changes in the material processing occur.
3. At least one item per two-month period.

If an interference appears, either a higher resolution detector should be acquired or an adequate peak-stripping routine applied. In both cases, additional standards that include the interfering radiations should be selected and the system should be recalibrated.

No item should be assayed if the measured response exceeds that of the highest enrichment standard by more than twice the standard deviation in the response from this standard.

## 6. ERROR ANALYSIS

A regression or analysis-of-variance technique should be used to determine the uncertainty in the calibration constants.

The measurement-to-measurement variance should be determined by periodically observing the net response from the standards and repeating measurements on selected process items. Each repeated measurement should be made at a different location on the container surface, at different times of the day, and under different ambient conditions.<sup>9</sup> The standard deviation should be determined and any trends (e.g., trends due to time or temperature) corrected for.

The item-to-item variance due to the variation in wall thickness should be determined. The variance in the container wall thickness should be determined from measurements of the sample container wall thickness, either during the course of the assays or from separate measurements of randomly selected samples. The computed variance in the samples should be used as the variance of wall thickness. This variance should be multiplied by the effect of a unit variation in that thickness on the measured 185.7-keV (see, e.g., Table 2) response to determine its contribution to the total measurement variance.

Item-to-item variations other than those measured, e.g., wall thickness, should be determined by periodically (see guidelines in Regulatory Position 5) selecting an item and determining the enrichment by an independent technique traceable to, or calibrated with, NBS standard reference material. A recommended approach is to adequately sample and determine the  $^{235}\text{U}$  enrichment by calibrated mass spectrometry. In addition to estimating the standard deviation of these comparative measurements, the data can also be used to verify the continued stability of the instrument calibration. If any significant deviation of the calibration is noted from these comparisons, the cause of the change should be identified before further assays are performed.

<sup>9</sup> The variance due to counting (including background) and the variance due to inhomogeneity, ambient conditions, etc., will be included in this measurement-to-measurement variance.



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Alvar, K., H. Lukens, and N. Lurie, "Standard Containers for SNM Storage, Transfer, and Measurement," U.S. Nuclear Regulatory Commission, NUREG/CR-1847, 1980. Available through the NRC/GPO Sales Program, U.S. Nuclear Regulatory Commission, Washington, D.C. 20555.

This report describes the variations of container properties (especially wall thicknesses) and their effects on NDA measurements. A candidate list of standard containers, each sufficiently uniform to cause less than 0.2 percent variation in assay results, is given, along with comments on the value and impact of container standardization.

Augustson, R. H., and T. D. Reilly, "Fundamentals of Passive Nondestructive Assay of Fissionable Material," Los Alamos Scientific Laboratory, LA-5651-M, Albuquerque, New Mexico, 1974.

This report contains a wealth of information on nondestructive assay techniques and their associated instrumentation and has an extensive treatise on gamma ray enrichment measurements.

Sher, R., and S. Untermeyer, "The Detection of Fissionable Materials by Nondestructive Means," American Nuclear Society Monograph, La Grange Park, Illinois, 1980.

This book contains a helpful overview of a wide variety of nondestructive assay techniques, including enrichment measurement by gamma ray spectrometry. In addition, it contains a rather extensive discussion of error estimation, measurement control techniques, and measurement statistics.

## VALUE/IMPACT STATEMENT

### 1. PROPOSED ACTION

#### 1.1 Description

Licensees authorized to possess at any one time more than one effective kilogram of special nuclear material (SNM) are required in § 70.51 of 10 CFR Part 70 to determine the inventory difference (ID) and the associated standard error (SEID) for each element and the fissile isotope of uranium contained in material in process. The determination is made by measuring the quantity of the element and of the fissile isotope for uranium.

It is not usually possible to determine both element and isotope with one measurement. Therefore, a combination of techniques is required to measure the SNM ID and the SEID by element and by fissile isotope. Passive gamma ray spectroscopy is a nondestructive method for measuring the relative concentration of the fissile isotope  $^{235}\text{U}$  in uranium. This technique is then used in conjunction with an assay for the element uranium to determine the amount of  $^{235}\text{U}$ .

Regulatory Guide 5.21 describes conditions for  $^{235}\text{U}$  enrichment measurements using gamma ray spectroscopy that are acceptable to the NRC staff. The proposed action will revise the guide to conform to current usage and to add information on the state of the art of this technique.

#### 1.2 Need

The proposed action is needed to bring Regulatory Guide 5.21 up to date.

#### 1.3 Value/Impact Assessment

##### 1.3.1 NRC Operations

The experience and improvements in technology that have occurred since the guide was issued will be made available for use in the regulatory process. Using these updated techniques should have no adverse impact.

##### 1.3.2 Other Government Agencies

Not applicable.

##### 1.3.3 Industry

Since industry is already applying the techniques discussed in the guide, updating these techniques should have no adverse impact.

##### 1.3.4 Public

No impact on the public can be foreseen.

#### 1.4 Decision on Proposed Action

The guide should be revised to reflect improvements in technique and to bring the guide into conformity with current usage.

### 2. TECHNICAL APPROACH

Not applicable.

### 3. PROCEDURAL APPROACH

Of the alternative procedures considered, revision of the existing regulatory guide was selected as the most advantageous and cost effective.

### 4. STATUTORY CONSIDERATIONS

#### 4.1 NRC Authority

Authority for the proposed action is derived from the Atomic Energy Act of 1954, as amended, and the Energy Reorganization Act of 1974, as amended, and implemented through the Commission's regulations.

#### 4.2 Need for NEPA Assessment

The proposed action is not a major action that may significantly affect the quality of the human environment and does not require an environmental impact statement.

### 5. RELATIONSHIP TO OTHER EXISTING OR PROPOSED REGULATIONS OR POLICIES

The proposed action is one of a series of revisions of existing regulatory guides on nondestructive assay techniques.

### 6. SUMMARY AND CONCLUSIONS

Regulatory Guide 5.21 should be revised to bring it up to date.