

August 1974

U.S. ATOMIC ENERGY COMMISSION

# REGULATORY GUIDE

DIRECTORATE OF REGULATORY STANDARDS

## REGULATORY GUIDE 5.37

### IN SITU ASSAY OF ENRICHED URANIUM RESIDUAL HOLDUP

#### A. INTRODUCTION

Part 70 of Title 10 of the Code of Federal Regulations requires each licensee authorized to possess more than 350 grams of contained U-235 to conduct a physical inventory of all special nuclear material in his possession at intervals not to exceed 12 months. Certain licensees authorized to possess more than one effective kilogram of special nuclear material are required to conduct measured physical inventories of their special nuclear materials more frequently than annually depending on the materials. Further, these licensees are required to conduct their nuclear material physical inventories in compliance with specific requirements set forth in Part 70. Inventory procedures acceptable to the Regulatory staff are detailed in Regulatory Guide 5.13, "Conduct of Nuclear Material Physical Inventories."

Enriched uranium residual holdup is defined as the inventory component remaining in and about process equipment and handling areas after those collection areas have been prepared for inventory. Whenever possible, process equipment should be designed\* and operated so as to minimize the amount of holdup. This guide describes procedures acceptable to the Regulatory staff for the in situ assay of the residual uranium holdup.\*\*

Assay information can be used in one of two ways:

1. When the limit of error of uranium holdup is compatible with the plant limit of error on material unaccounted for (LEMUF), the material balance can be computed using the measured contents of uranium

\* Design features to minimize holdup in process equipment are the subject of a series of Regulatory Guides.

\*\* Assay of residual plutonium holdup is the subject of Regulatory Guide 5.23, "In Situ Assay of Plutonium Residual Holdup."

holdup. Additional cleanout and recovery for accountability will then not be necessary.

2. When the limit of error of uranium holdup is not compatible with the plant LEMUF, the information obtained in the holdup survey can be used to locate principal uranium accumulations. Once located, substantial accumulations can be recovered, transforming the uranium to a more accurately measurable inventory component. Having reduced the amount of uranium holdup, the limit of error on the remeasurement of the remaining holdup may be sufficiently reduced to be compatible with overall plant LEMUF requirements.

In situ assay guides describe methods to ensure that a measured value of residual holdup is included in each material balance. Because of unique measurement considerations, the procedures described in this guide for calibration and error evaluation are not consistent with general Regulatory guidance on those topics.

#### B. DISCUSSION

Uranium accumulates in cracks, pores, and zones of poor circulation within and around process equipment. The walls of process vessels and associated plumbing often become coated with uranium during processing of solutions. Uranium also accumulates in air filters and associated ductwork. The absolute amounts of uranium holdup must be small for efficient processing and proper hazards control. However, the total amount of uranium holdup may be significant in the context of the plant material unaccounted for (MUF).

The measurement procedures detailed in this guide are based on the controlled observation of gamma rays which are spontaneously emitted in association with the decay of U-235. To accomplish the gamma ray assay, it is essential to consider the facility in terms of a series of zones which can be independently assayed. Such zones are designated as "collection zones."

#### USAEC REGULATORY GUIDES

Regulatory Guides are issued to describe and make available to the public methods acceptable to the AEC Regulatory staff of implementing specific parts of the Commission's regulations, to delineate techniques used by the staff in evaluating specific problems or postulated accidents, or to provide guidance to applicants. Regulatory Guides are not substitutes for regulations and compliance with them is not required. Methods and solutions different from those set out in the guides will be acceptable if they provide a basis for the findings requisite to the issuance or continuance of a permit or license by the Commission.

Published guides will be revised periodically, as appropriate to accommodate comments and to reflect new information or experience.

Copies of published guides may be obtained by request indicating the divisions desired to the U.S. Atomic Energy Commission, Washington, D.C. 20545, Attention: Director of Regulatory Standards. Comments and suggestions for improvements in these guides are encouraged and should be sent to the Secretary of the Commission, U.S. Atomic Energy Commission, Washington, D.C. 20545, Attention: Chief, Public Proceedings Staff.

The guides are issued in the following ten broad divisions:

- |                                   |                        |
|-----------------------------------|------------------------|
| 1. Power Reactors                 | 6. Products            |
| 2. Research and Test Reactors     | 7. Transportation      |
| 3. Fuels and Materials Facilities | 8. Occupational Health |
| 4. Environmental and Siting       | 9. Antitrust Review    |
| 5. Materials and Plant Protection | 10. General            |

## 1. DELINEATION OF COLLECTION ZONES

Each uranium processing facility can be conceptually divided into a series of contiguous collection zones. Individual process machines, air filters, and separated item areas that can be isolated from other activities may be suitable discrete collection zones. For assay purposes, the dimensions of a discrete collection zone may not be compatible with assay accuracy objectives. In such cases, each discrete zone can be further subdivided as described in Section B.4.3 of this guide.

## 2. GAMMA RAY HOLDUP ASSAY

Two considerations are critical to the holdup assay. First, to perform an assay, the U-235 gamma rays must reach the detector and be detected. Second, the observed response must be attributable to the collection zone being assayed. Therefore, the assay scheme is calibrated to compensate for the poor penetrability of the U-235 gamma ray. Also, the detector is collimated to separate a collection zone from its neighboring zones and from the radiation background.

For each gram of U-235, 185.7-keV gamma rays are emitted at a rate of  $4.3 \times 10^4$  per second. This gamma ray is the only radiation emitted in the decay of U-235 that is useful for this assay application. Unless mixed with plutonium or thorium, all other gamma rays are usually attributable to the Compton scattering of high-energy gamma rays emitted by the Pa-234 daughter of U-238. The background at 185.7 keV due to this source of radiation varies depending on the length of time between the separation of the U-238 daughters from the uranium (as frequently occurs during conversion processes) and the assay. This interference is very important for low-enrichment uranium, but much less so at very high-enrichment values.

On uranium recycled after exposure in a nuclear reactor, a sufficient quantity of U-232 or U-237 may be present to emit a measurable amount of interfering gamma radiation.

When uranium is mixed with thorium, the background gamma ray spectrum becomes much more complex. The background spectrum may vary because the daughters of thorium-232 can be volatilized to different extents during typical fuel processing.

To accomplish gamma ray assay of enriched uranium holdup, it is necessary to know from separate measurements the enrichment of the uranium holdup and to measure the emitted 185.7-keV gamma ray with sufficient resolution to enable the intensity of that gamma ray to be determined in the presence of the interfering radiations encountered.

## 2.1 Gamma Ray Detection Instruments

Data processing electronics include a single-channel analyzer for the 185.7-keV photopeak, a timer-scaler unit, and a second single-channel analyzer used to determine the background radiation correction. Battery-powered gamma ray analysis systems suitable for this application are commercially available and can enhance operational convenience (Ref. 1). Methods for determining energy window settings are provided in References 2 and 3.

The detection efficiency and resolution of NaI(Tl) detectors are generally adequate for this application when the uranium is not mixed with plutonium or thorium. Cadmium telluride (Refs. 4 and 5) has better resolution than NaI and may prove adequate to resolve the 185.7-keV gamma ray from thorium or plutonium gamma rays. Ge(Li) and intrinsic germanium semiconductor gamma-ray detectors have very high resolution but are less efficient than the other detector types and are more difficult to operate and maintain.

Detector dimensions are selected to provide a high probability for detecting the 185.7-keV gamma ray. The crystal depth is chosen so that most of the 185.7-keV gamma rays striking the crystal will lose all their energy within the crystal.

## 2.2 Collimators for Gamma Rays

A shaped shield constructed of any dense nonradioactive material is appropriate for gamma ray collimation. More than 98% of all 185.7-keV gamma rays striking a 0.35-cm-thick sheet of lead are absorbed or scattered.

The collimator will be most effective when it is concentric about the crystal and, for NaI detectors, the photomultiplier and the photomultiplier base. Extending the collimator forward of the crystal at least a distance equal to half the diameter of the crystal, and preferably the full diameter, is recommended (Refs. 2 and 3). Making this distance adjustable to reproducible settings will facilitate use of the collimated detector for a range of collection zone sites.

## 2.3 Check Source for Gamma-Ray Assay

It is important to check the operation of the detection system prior to each inventory sequence. An appropriate check source enables the stability of the assay instrument to be tested at any location. Such a source can be prepared by implanting a small encapsulated uranium source (containing ~0.5 g U) in the face of a plug of shielding material. The plug is shaped to fit and close the collimator channel, and the source is positioned to be adjacent to the crystal when

the plug is in place. When the response from the check source remains within the expected value, the previous calibration data are assumed to be valid. If not, the energy window may have shifted, or the unit may be in need of repair and recalibration.

#### 2.4 Calibration Source for Gamma Ray Assay

In situ holdup assay is essentially a problem of measuring the gamma ray emissions from uranium distributed in diverse patterns which may differ widely in subsequent assays of the same collection zone. The diverse distribution of uranium is accompanied by significant variations in counting geometry and in the attenuation of the 185.7-keV gamma rays. For these reasons, routine calibration is based on a feedback model described in Section B.5.3.2 of this guide. For initial assay operations and to verify the consistency of a previous calibration, a single calibration standard is used. A single calibration standard is contrary to general Regulatory guidance on the sufficiency of such standards; in this application, however, additional standards provide essentially no additional useful information, because geometric variations and differences in attenuation are typically the dominant sources of error. Therefore, using one standard for calibration, the assay response can be assumed to be directly proportional to the U-235 content. The proportionality ratio of net counts per gram of U-235 is calculated by subtracting the background counts from the counts due to the calibration standard and dividing by the U-235 mass of the calibration standard. This procedure is acceptable for initial operations only, until calibration can be based on holdup recovery data.

The observed assay response is compared to the response obtained when the zone contains a known amount of uranium. The response is assumed to be linearly proportional to uranium content, after corrections for attenuation are made (Ref. 5). To be representative of typical holdup situations, a calibration standard is prepared consisting of a uranium metal disk or an encapsulated  $\text{UO}_2$  disk with a bed thickness of less than 0.2 cm. Care must be exercised in the preparation of the calibration standard to ensure that the amount of total uranium and U-235 encapsulated is known. It is important to measure the gamma ray attenuation through the encapsulating material and correct the calibration standard response to compensate for that attenuation. The amount of uranium encapsulated in the gamma ray calibration standard is selected to be representative of typical accumulations.

### 3. ISOLATION OF COLLECTION ZONES

To ensure that each collection zone is independently assayed, it is necessary to screen all radiations from the detector except those radiations emanating from the collection zone being assayed. This is principally accomplished through the use of the collimators

described in Section B.2.2. Two additional means exist to further isolate a collection zone.

#### 3.1 Detector Positioning

When uranium is located in back of the zone under assay in another collection zone or in a storage facility, the detector can be positioned between the radiation sources or above or below the collection zone to isolate the zone for assay.

#### 3.2 Shadow Shielding

When it is not possible to avoid interfering radiations through the collimator design or through choosing the detector position for assay, it may be possible to move a shield panel between the source of interfering radiations and the collimator zone under assay. If the shield panel is sufficiently thick and its dimensions match or exceed the back side of the collection zone under assay, no interfering radiations will penetrate through the shadow shield to the detector. A lead sheet 0.4 to 0.5 cm thick, which might be mounted on wheels as an upright panel, is generally adequate for this application.

### 4. CALIBRATION OF COLLECTION ZONES

Each collection zone is independently calibrated, as background factors and the composition of each zone vary widely from zone to zone. A collection zone is initially calibrated through the in situ measurement of a known calibration standard with a correction for attenuation. When such a program is not possible, the initial calibration can be based on the calculation of the anticipated response or through measuring a mockup of the collection zone of interest (Refs. 3, 6). The limit of error of the assay is then estimated through determining the response variation over the range of each parameter affecting the measurement.

The calibration obtained through this procedure is recommended until a history of comparisons between predicted and recovered holdup quantities is developed, as described in Section B.5 of this guide.

#### 4.1 Detector Positioning

On the basis of a detailed examination of the physical layout of the facility, some preliminary measurements are made to determine optimum detector positions for holdup assay. Once the assay positions for the detector and shadow shields are established, permanently marking the assay positions will ensure the reproducibility of subsequent measurements.

#### 4.2 Calibration Sources

Since this assay is to measure the amount of uranium holdup, it is appropriate to use uranium as the calibration standard material. Further, as the uranium holdup will generally be distributed over a large surface area, it is recommended that the gamma ray calibration standard be fabricated to resemble this characteristic, as described in Section B.2 of this guide.

### 4.3 Calibration Procedures

Once the principal items containing uranium have been removed and the detector located in its assay position, the response to a calibration standard combined with the uranium already held up is obtained. When the collection zone is appropriately isolated, two factors influence the observed response to the calibration standard:

1. The location of the calibration standard within the collection zone, and
2. The shielding of radiations from the calibration standard caused by the items comprising the collection zone.

The geometric response variation is measured by observing the response to the calibration standard with the standard positioned in various parts of the collection zone, avoiding internal items which may strongly attenuate the radiation emanating from the standard. When the collection zone consists of a hollow box, pipe, or duct, attenuation is either relatively uniform or negligibly small. Reference 7 describes procedures for the assay of columns or solution containers. The calibration of each collection zone then becomes a matter of appropriately averaging the geometric response variations (Ref. 3). The average response for the entire collection zone is assumed to properly represent that zone. If, however, it is known that uranium accumulates in one particular location within a collection zone, the response to the standard is

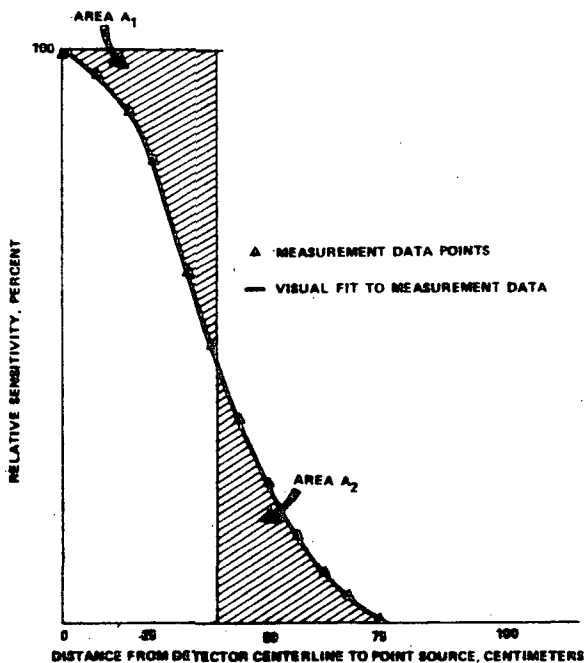


FIGURE B-1 EQUIVALENT DIAMETER SUBZONE TO ACHIEVE A FLAT PLANAR RESPONSE. SELECT D SUCH THAT AREA  $A_1 = A_2$ .

emphasized when the standard is located near the principal collection site.

If the item to be assayed consists of a large unit, assay performance may be enhanced by subdividing the unit into smaller contiguous measurement zones (Ref. 3). The repeat dimensions of the subzones are determined by measuring the response while moving the standard along an axis perpendicular to the detector centerline. By studying the response curve, the distance D is selected as the point beyond which sufficient activity is detected to flatten the response within the subzone. Each subzone will measure 2D across its face. As illustrated in Figure B-1, the response about the centerline is assumed to be symmetrical and only half the traverse is indicated. In Figure B-1, D is selected such that the area under the curve to the right of D is approximately equal to the area above the curve to the left of D ( $A_1 \approx A_2$ ). Note: the distance from the collection zone to the detector, or the distance from the crystal face to the end of the collimator, or both, can be varied to divide the collection zone into an integral number of subzones.

To use this relationship, the detector is first positioned at point d and a reading is taken. Point d is the center of the first subzone, selected to coincide with the physical edge of the calibration zone. The detector is then moved a distance 2D along the traverse to the center point of the second subzone, and the second measurement taken. The cycle is repeated to include all of the larger collection zone. The value interpreted for calibration for each subzone corresponds to the maximum of the traverse across each subzone because the response has been flattened. The content of the entire collection zone is the sum of the contributions from the subzones.

### 5. ESTIMATION OF THE HOLDUP UNCERTAINTY

The random uncertainty components in this application are frequently negligible in comparison with the geometric uncertainty, attenuation, and, in some cases, uncertainty in the U-235 enrichment. When initiating this assay program, it is appropriate to estimate the assay uncertainty components by assuming the measured range ( $R_i$ ) of the  $i^{\text{th}}$  fluctuation constitutes an interval with a width of four standard deviations, ( $s_i$ ). The midpoint of the range estimates the mean effect (Ref. 8).

$$s_i^2 = (R_i/4)^2 \quad (1)$$

#### 5.1 Response Uncertainties

##### 5.1.1 Counting Statistics

The magnitude of the uncertainties attributable to variations in the geometric distribution and in the

attenuation of the radiations are expected to dominate the total response uncertainty. The relative standard deviation due to counting statistics can usually be made as small as desired (1) through using more efficient detectors or (2) by extending the counting period. Having 1,000 to 10,000 net counts is generally sufficient for most holdup assay applications.

### 5.1.2 Instrument Instabilities

Fluctuations in ambient temperature, humidity, electronic noise, and line voltage (for non-battery-powered electronic units) generally affect the stability of electronic systems. The magnitude of this uncertainty can be estimated by monitoring the check standard response and determining the range of variability as described in Section B.5 of this guide.

### 5.1.3 Geometric Uncertainty

The geometrical variation in the observed response is measured by moving the calibration source within the bounds of each collection zone. Two cases are described.

#### 5.1.3.1 Isolated Collection Zones

When a single unit comprises a collection zone, the standard is moved to all sites within the zone at which an accumulation of uranium might occur. With sufficient collimation, the response for the collection zone under investigation is independent of its neighboring zones. The average of the response, weighted to reflect pre-judgements on the likelihood of accumulation sites, is then used as the calibration point. As shown in Section 5, the range of values can be assumed to comprise an expectation interval four standard deviations wide. The geometric uncertainty is then estimated using Equation 1.

#### 5.1.3.2 Overlapping Collection Zones

When a collection zone is subdivided into overlapping subzones, the geometric uncertainty due to the dimension perpendicular to the detector collection zone centerline is eliminated through the area-averaging calibration method described in Section B.4.3. The uncertainty in the depth dimension in each subzone can be determined through the procedure outlined for isolated collection zones. Judgment can be used to weight the calibration data to emphasize principal accumulation sites.

### 5.1.4 Attenuation Uncertainty

To obtain useful assay results by detecting the 185.7-keV gamma ray, it is necessary to correct each assay for attenuation of the signal either within the uranium holdup material or by structural materials. Without this critical correction, the assay is essentially worthless. Details for establishing an appropriate

attenuation correction are given in Laboratory Exercise #4 of Reference 3.

## 5.2 Interpretation Uncertainties

### 5.2.1 Calibration Standard

The calibration standard may be fabricated of uranium that differs from the uranium holdup in enrichment, chemical form, matrix, and total quantity. During initial operations, these potential sources of bias can be compensated by calculating the change in response due to each variation or by measuring the response variations through appropriate experimental mockups. When the assay is calibrated based on holdup recovery data, the effects of these error sources will be included in the calibration and error estimation.

### 5.2.2 Interfering Radiations

An uncertainty in the observed gamma ray response may arise due to the presence of extraneous gamma ray emitters or due to fluctuations in the background from the Compton scattering of higher-energy gamma rays. In particular, when uranium is mixed with thorium or plutonium, special precautions must be taken to compensate for variable blend ratios and the disrupted equilibrium of radioactive daughter products. Extraneous low-energy radiations can be suppressed by covering the crystal face with an appropriate filter (e.g., 0.75mm of cadmium).

### 5.2.3 Enrichment Uncertainty

If the process equipment is thoroughly cleaned each time the enrichment of the uranium feed is changed, the holdup will consist primarily of the current material. New calibration standards can be prepared or the previous yield data can be normalized to correct for enrichment changes. When mixing occurs, use of the stream-averaged enrichment is appropriate. The uncertainty bounds are estimated by considering the batches of highest and lowest enrichment and computing the corresponding range.

## 5.3 Holdup and Its Associated Uncertainty

### 5.3.1 Initial Operations

During the initial phase of operations, the error associated with the in situ assay of uranium holdup is estimated as the square root of the combined component mean-square uncertainties determined in Sections B.5.1 and B.5.2.

### 5.3.2 Routine Operations

To ensure the validity of assay predictions and to realistically estimate the uncertainty in those predictions, the amount of uranium recovered when a

collection zone is cleaned out can be used. By comparing the amount of uranium recovered to the recovery amount predicted through the in situ assay, the collection zone calibration is updated and the assay uncertainty is based on relevant data.

The update data is computed as the difference in the assays before and after cleanout:

$$U_{\text{assay}} = R_{\text{before}} - R_{\text{after}} \quad (2)$$

The difference ( $\Delta$ ) in assay and recovery,

$$\Delta = U_{\text{assay}} - U_{\text{recovery}} \quad (3)$$

is then computed.

The standard deviation in the  $\Delta$  values ( $s_{\Delta}$ ) is computed separately for each collection zone, including no more than the twelve preceding measurement tests:

$$s_{\Delta} = \left[ \left( \sum_{i=1}^k \Delta_i^2 - \left( \sum_{i=1}^k \Delta_i \right)^2 / K \right) / (K-1) \right]^{1/2} \quad (4)$$

When a value of  $\Delta$  is determined, it is used to update the estimate  $s_{\Delta}$ . The standard deviation estimate  $s_{\Delta}$  can be used to estimate the systematic error in the assay prediction for the collection zone for which it has been established.

The amount of uranium collected during the cleanout of a specific collection zone can be assayed through sampling and chemical analysis, or through other applicable nondestructive assay methods.\*

## C. REGULATORY POSITION

To develop a program for the periodic in situ assay of enriched uranium residual holdup as an acceptable measurement method for this inventory component, it is necessary to consider the factors in the following sections.

### 1. DELINEATION OF ASSAY COLLECTION ZONES

A plan of a uranium processing facility should be examined to establish independent collection zones.

1. Assay site(s) should afford a clear, unobstructed view of the collection zone with no other collection or storage areas in the line of sight of the collimator assembly. The location of the detector probe above or below the collection zone should be considered if an unobstructed side view is not possible. If an unobstructed view is not possible, shadow shielding should be used to isolate the collection zone for assay.

2. The assay site should be set back as far as possible from each collection zone to reach a compromise between interference from neighbor zones and efficient counting.

3. Each assay site should be marked with paint or colored tape on the floor to enable reproducible assays. The height setting for midpoint assay should be recorded in the measurement log corresponding to each assay site.

## 2. ASSAY SYSTEM

### 2.1 Detector Selection

Nal(Tl) detectors are generally suitable for this application when the uranium is not mixed with thorium or plutonium. The crystal depth should be sufficient to detect a significant percentage of 185.7-keV gamma rays. For Nal(Tl), a one-inch depth is recommended. Cadmium telluride, Ge(Li), or intrinsic germanium detectors should be used when Nal resolution is inadequate to separate the U-235 activity from interfering radiations.

The crystal should be stabilized with a suitable radioactive source. An internal CsI seed containing Am-241 is recommended for Nal applications. The electronics should be capable of stabilizing on the reference radiation emitted by the seed.

Two single-channel analyzers should be provided with lock-set energy windows. One channel should be set to admit the 185.7-keV gamma rays from U-235. The second channel should be set above the first window to provide a background correction for the assay window. The electronics unit should have a temperature stability of less than 0.1% per °C.

### 2.2 Gamma Ray Collimator

A cylinder of shielding material such as lead should be made coaxial with the gamma ray detector. The end of the cylinder opposite the crystal should be blocked with the shielding material. The thickness of the collimator should be chosen to provide sufficient directionality for the specific facility (0.35 cm of lead thickness should be sufficient for most applications). The collimator sleeve should be adjustable over the end of the crystal to reproducible settings to vary the degree of collimation for different collection zones.

### 2.3 Gamma Ray Check Source

An encapsulated uranium check source should be provided. The source should be small enough to be

---

\*See Regulatory Guide 5.11, "Nondestructive Assay of Special Nuclear Materials Contained in Scrap and Waste."

implanted in a section of shielding material so shaped as to close off the collimator opening. The check source should be positioned adjacent to the detector. The source should contain an amount of uranium sufficient to provide a gross count rate of 1,000 to 10,000 counts per second in the energy region of interest.

## 2.4 Gamma Ray Calibration Source

A calibration standard should be fabricated by encapsulating  $\text{UO}_2$  or U metal in a disk. The total amount of uranium encapsulated should be closely monitored. Attenuation losses within the bed of  $\text{UO}_2$  or U metal and through the encapsulating material should be measured or computed and the calibration standard response normalized to counts per gram with these corrections incorporated.

## 3. CALIBRATION

Each collection zone should be independently calibrated when all in-process material has been located so that it will not influence the response to the calibration standards.

### 3.1 Instrument Check

The stability of the gamma ray detection system should be tested prior to each inventory. If the check source measurement is consistent with previous data (i.e., is within plus or minus two single-measurement standard deviations of the mean value of previous data), previously established calibration data should be considered valid. If the measurement is not consistent, the operation of the unit should be checked against the manufacturer's recommendations and repaired and/or recalibrated, as required.

### 3.2 Zone Calibration

The geometric response profile for each collection zone should be determined by measuring the variation in the response as a calibration standard is moved within the defined limits of the collection zone, correcting for attenuation at each point. The response variation should then be averaged to determine the response per gram of uranium for that collection zone. The averaging should be weighted to reflect known local accumulation sites within each collection zone. The response per gram should be used to directly translate the observed response to grams of uranium after the response is corrected for background.

#### 3.2.1 Subzone Calibration

When a collection zone is too large to be accurately measured in a single assay, the collection zone should be

divided into overlapping subzones. The repeat dimensions of each subzone perpendicular to the detector-to-collection-zone line should be determined so that the response variation across that distance is nulled. The residual geometric uncertainty should be determined by measuring the response as a calibration standard is moved along the depth coordinate, correcting for attenuation. The calibrated response should then reflect the average of the depth response, weighted to reflect known accumulation sites.

## 4. ASSAY PROCEDURES

### 4.1 Assay Log

An assay log should be maintained. Each collection zone or subzone should have a separate page in the assay log, with the corresponding calibration derived on the page facing the assay data sheet. Recording space should be provided for the date of measurement, gross counts, corrected counts, and the corresponding grams of uranium from the calibration in addition to position and instrument electronic setting verification.

### 4.2 Preassay Procedures

Prior to inventory, the enrichment of the uranium processed during the current operational period should be determined. Variations in the gamma ray yield data from the calibration standard should be calculated. Either the calibration data or the predicted holdup should then be corrected to reflect this error.

Prior to each inventory, the operation of the gamma-ray assay detection systems should be checked.

Prior to any assay measurements, feed into the process line should be stopped. All in-process material should be processed through to forms amenable to accurate accountability. All process, scrap, and waste items containing uranium should be removed from the process areas to approved storage areas to minimize background radiations.

### 4.3 Measurements

Before assaying each collection zone, the operator should verify that floor location, probe height, and electronics settings correspond to previous measurements. All check and calibration sources should be sufficiently removed so as not to interfere with the measurement. Prior to taking a measurement, a visual check of the zone and the line of sight of the detector probe should be made to assure that no obvious changes have been made to the process area and that no unintended accumulations of uranium remain within the collection zone. The operator should initial the measurement log to assure compliance for each collection zone.

When all the preceding steps have been completed, the measurement at each site should be taken and recorded. An attenuation correction measurement should be made and the corrected response should be converted to grams of uranium. If a high response is noted, the cause should be investigated. If the collection zone contains an unexpectedly large content of uranium, that collection zone should be cleaned to remove the accumulation for conversion to a more accurately accountable material category. After the cleanout has been completed, the zone should be re-assayed and the recovered material quantity used to test the validity of the zone calibration.

## 5. ESTIMATION OF THE HOLDUP UNCERTAINTY

During the initial implementation of this program, the error quoted for the holdup assay should be computed on the basis of estimating the uncertainty components as described in Sections B.5.1 and B.5.2 of this guide.

Prior to the cleanout of any collection zone for whatever purpose, that zone should be prepared for

assay and measured as described in Section C.4 of this guide. Following this assay the collection zone should be cleaned out and the collected uranium should then be assayed using an appropriately accurate assay method. When the collection zone has been cleaned and the collected uranium removed, the collection zone should be re-assayed. The recovered uranium should be used to update the calibration and, from the sixth test on, should serve as the assay uncertainty estimate. Separate records should be maintained for each collection zone to estimate the uncertainty in assaying the uranium holdup.

During each physical inventory, the calibration in at least 10% of all collection zones should be updated. The zones should be selected in such a manner that all collection zones are updated in ten consecutive physical inventories. In small plants with less than ten collection zones, at least one zone should be updated during each physical inventory.

To ensure that error predictions remain current, only data of the twelve preceding independent tests should be used to estimate the assay uncertainty.

## REFERENCES

1. L. A. Kull, "Catalogue of Nuclear Material Safeguards Instruments," BNL-17165 (August 1972).
2. R. B. Walton, et al., "Measurements of UF<sub>6</sub> Cylinders with Portable Instruments," Nucl. Technol., 21, 133 (1974).
3. R. H. Augustson and T. D. Reilly, Editors, "A Training Manual for Nondestructive Assay with Portable Instrumentation," LASL Report LA-UR-73-1525 (1974).
4. W. Higinbotham, K. Zanio, and W. A. Kutagawa, "CdTe Gamma Spectrometers for Nondestructive Analysis of Nuclear Fuels," IEEE Trans. Nucl. Science 20, 510 (1972).
5. F. Wald, R. O. Bell, H. B. Serreze, "Cadmium Telluride Detectors—Second Interim Technical Report," Report C00-3545-1, July 1972. Available through National Technical Information Service, U.S. Department of Commerce, Springfield, Virginia 22151.
6. W. D. Reed, Jr., J. P. Andrews, and H. C. Keller, "A Method for Surveying for Uranium-235 with Limit of Error Analysis," Nucl. Mat. Mgmt. 2, 395 (1973).
7. M. M. Thorpe, R. B. Walton, and L. V. East, "Assay of Uranium Solution Storage Tanks," LASL Report LA 4794-MS (1971), pp. 14.
8. M. G. Natrella, "Experimental Statistics," National Bureau of Standards Handbook 91, U.S. Department of Commerce (1966). (See Section 2-2.4.)