

WITHDRAWN

February 1975

U.S. NUCLEAR REGULATORY COMMISSION

# REGULATORY GUIDE

## REGULATORY GUIDE 5.47

### CONTROL AND ACCOUNTABILITY OF PLUTONIUM IN WASTE MATERIAL

#### A. INTRODUCTION

Section 73.60, "Additional Requirements for the Physical Protection of Special Nuclear Material at Fixed Sites," of 10 CFR Part 73, "Physical Protection of Plants and Materials," requires that certain licensees search each package leaving a material access area\* for concealed special nuclear material to prevent plutonium from being removed from the licensee's control. Physical search procedures or equipment capable of detecting concealed plutonium may be used. Nondestructive search techniques such as gamma ray spectrometry and neutron assay are recommended when used with a tamper-safing system to ensure that no concealed plutonium is removed from a material access area in waste containers.

Section 70.51, "Material Balance, Inventory, and Records Requirements," of 10 CFR Part 70, "Special Nuclear Material," requires certain licensees to conduct physical inventories at bimonthly intervals. On the basis of each bimonthly inventory, those licensees are required to calculate a material balance, including the material unaccounted for (MUF) and its associated limit of error (LEMUF). Discards of contaminated waste must be included in the material balance. Section 70.51 further states that the LEMUF must not exceed specified limits. With proper controls, nondestructive assay (NDA) techniques may be applied to the assay of plutonium-contaminated waste material.

This guide describes procedures which are acceptable to the NRC staff for the control and accountability of plutonium-contaminated waste. Specifically, this guide describes procedures for complying with paragraph 73.60(b) for searching plutonium-contaminated waste packages for concealed plutonium. In addition, this

guide describes procedures for complying with assay requirements related to plutonium-contaminated waste given in paragraph 70.51(e)(4)(i).

#### B. DISCUSSION

A variety of materials become contaminated during the processing of plutonium. To be economically recoverable, the contaminated material must contain enough plutonium to offset the scrap recovery costs. Materials containing very small quantities of plutonium, or process materials from which the plutonium cannot be economically separated, are often considered as process waste. Provisions relating to the disposition of such materials are contained in 10 CFR Part 20.

As noted in the introduction, the materials protection interest in such materials is twofold. First, to prevent significant quantities of plutonium from being concealed in containers, which would permit plutonium to be removed from the licensee's control. Second, to include in the measured material balance, all plutonium contained in waste. The same measurement systems and operations are able to search packages for substantial quantities of concealed plutonium and to assay contaminated waste for plutonium content. Thus, both of these considerations are addressed in this guide.

#### 1. Container Selection

Plutonium-contaminated waste is typically packaged in 55-gallon drums for storage or shipment. It is often packaged in small primary containers (typically 4-6 liters capacity) first and then combined in large containers to reduce the waste-handling problem.

It is desirable to search and assay the waste in small containers rather than in large containers for the following reasons:

1. High concentrations of plutonium in waste material can be identified more easily when small containers are assayed. The plutonium can then be recovered rather

\*"Material access area," as defined in 10 CFR Part 73, §73.2, "means any location which contains special nuclear material, within a vault or a building, the roof, walls, and floor of which each constitute a physical barrier."

#### USNRC REGULATORY GUIDES

Regulatory Guides are issued to describe and make available to the public methods acceptable to the NRC 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. Nuclear Regulatory Commission, Washington, D.C. 20555. Attention: Director of Standards Development. Comments and suggestions for improvements in these guides are encouraged and should be sent to the Secretary of the Commission, U.S. Nuclear Regulatory Commission, Washington, D.C. 20555. Attention: Docketing and Service Section.

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            |

than discarded, thus reducing the waste disposal problem.

2. The use of small containers would improve the assay accuracy and thus lower the contribution of waste to the MUF and LEMUF.

3. Assaying small containers of waste, each from a localized area, simplifies the calculation of individual area material balances (see Regulatory Guide 5.26, "Selection of Material Balance Areas and Item Control Areas").

4. Use of small containers facilitates the comparative assay procedures which are recommended to generate current estimates of the systematic assay error. Fabricating calibration standards and testing the performance of the instruments is made easy by assaying small containers.

5. It is more difficult to conceal material in small containers than in large containers. Therefore, assay of the small containers, combined with an effective tamper-safing program, would enhance the ability to ensure that waste packages are not used to conceal plutonium to remove it from the site.

Since the same volume of waste can be packaged in long, narrow-diameter cylindrical containers as in short, wide-diameter containers, narrow-diameter containers should be preferred because they can be assayed more accurately than wide-diameter ones (as discussed in Regulatory Guide 5.11, "Nondestructive Assay of Special Nuclear Material Contained in Scrap and Waste").

The size of the small containers and the material from which they are constructed are important factors. From the materials protection viewpoint, the most important factor in selecting and packaging the primary containers is the requirement that enough of the radiations emitted throughout each container must be detected to ensure that there are no blind spots. Such blind spots may conceal plutonium and would introduce large biases into the assay.

## 2. Segregation of Waste Materials

Plutonium spontaneously emits gamma rays and neutrons. Gamma rays are highly attenuated by heavy elements or densely compacted light elements. Neutrons, on the other hand, penetrate quite well through most heavy elements but are strongly attenuated by light elements.

Waste materials often consist of contaminated hydrogenous materials like paper wipes and plastics. Water is often present, the amount varying.

Different types of waste are often separately packaged for subsequent disposal. To achieve the desired search and accountability performance, waste which can be assayed using gamma ray methods should be packaged separately from waste which should be assayed using neutron methods.

## 3. Packaging and Sealing

For effective control and accurate accounting of plutonium-contaminated waste, waste should be packaged and sealed at the point where it is collected. Typically, contaminated waste is generated in cleanup or maintenance operations. It is collected and then transferred to a point where it can be removed ("bagged-out") from a glovebox line. Waste is often packaged in cardboard cylindrical containers ("ice cream containers") which are then individually heat-sealed in transparent polyethylene bags which prevent contamination after their transfer outside the glovebox enclosures. It is recommended that pressure-sensitive seals\* be applied on the next to the outermost heat-sealed plastic bag across each opening. Following assay and search, the package can be handled under item-control procedures.\*\* Comparable procedures are necessary for waste materials that cannot be packaged in this manner.

## 4. Assay and Search Measurements

The penetrability of gamma rays through a waste container can be determined by measuring the response to an external source of gamma rays. That response is measured under two conditions—first with the waste container not located in the vicinity of the instrument and then with the container positioned between the gamma ray source and the detector. The percent transmission (100 x container present/container absent) provides an indication of whether gamma ray assay is appropriate or whether neutron assay must be used. The percent transmission is also used to correct the gamma ray assay for internal attenuation, thereby improving the assay accuracy.

### 4.1 Gamma Ray Assay

The application of gamma ray spectrometry to plutonium assay is described in a report published by the Los Alamos Scientific Laboratory (ref. 1). A bibliography of other relevant references is also provided in that report, together with a description of the necessary theory, instrumentation, and data analysis procedures. The report also describes procedures to determine a gamma ray attenuation correction, container rotation and vertical scanning, and guidance on material categorization and packaging. Guidelines on the calibration of gamma ray waste assay are included in the LASL report, in ANSI Standard N15.20, "Guide to Calibrating Non-

\*See Regulatory Guide 5.10, "Selection and Use of Pressure-Sensitive Seals on Containers for Onsite Storage of Special Nuclear Material."

\*\*See 10 CFR Part 70, §70.51.

destructive Assay Systems,"\* and in a regulatory guide in preparation, "Calibration and Error Estimation Methods for Nondestructive Assay."

#### 4.2 Neutron Assay

Neutrons are spontaneously emitted in the decay of plutonium when the even-even isotopes (plutonium-238, 240, and 242) decay by spontaneous fission. Neutrons may also be emitted when alpha particles, emitted either in the decay of the plutonium isotopes or their daughter products, strike certain light nuclei. Neutron yield information is given in Regulatory Guide 5.23, "In Situ Assay of Plutonium Residual Holdup." Large errors in gross neutron assay can arise (1) when the isotopic composition changes, (2) when the concentration of high-yield ( $\alpha$ , n) target materials changes, and (3) when large differences occur in the amount or distribution of neutron-moderating materials. In assay applications requiring higher accuracy, spontaneous fission events are detected by the coincident detection of two or more of the radiations emitted in that type of reaction.\*\*

In the present application, high-density materials (i.e., materials having a gamma ray transmission of 1% or less at 414 keV) are examined by neutron assay.

It is necessary to establish a threshold sensitivity for detecting plutonium present for all types of waste materials. Items for which the assay indicates less than a statistically significant quantity (i.e., the detection threshold) are assumed (for material balance accounting) to contain no plutonium. Those items are not factored into the LEMUF calculation.

When the waste consists of machine parts, pipes, tools, etc. that have surface contamination, it may be necessary to disassemble or cut the item into small pieces. Such items are cleaned by appropriate methods (e.g., brushing, chipping, and acid leaching) to remove as much plutonium as possible prior to disposal.

#### 5. Homogeneous Waste Materials

Waste materials consisting of relatively homogeneous process residues can be searched using the procedures described above. However, more accurate assays may be made of homogeneous waste materials by traditional sampling and chemical analysis, coupled with a determination of the bulk quantity present.

#### 6. Abundance of Plutonium Isotopes

The assay for plutonium by gamma ray spectrometry is based on observation of one or, at most, a few

of the gamma rays emitted during the radioactive decay of plutonium-239. Therefore, it is also necessary to verify or measure the abundance of the isotope Pu-239 relative to the total plutonium to account for the quantity of materials by element, as required in 10 CFR Part 70.

Gamma ray spectrometry can be used to measure the relative abundances of the plutonium isotopes if they are unknown (ref. 1). When waste material is separated into single plutonium isotopic blends, gamma ray spectrometry can be used to verify a prior measurement of the isotopic abundances of the batch. Isotopic abundances can be verified by measuring the ratio of the intensity of gamma rays from two or more plutonium isotopes during waste assay (ref. 1).

#### 7. Instrument Shielding

The amount of plutonium contained in typical packages of waste will be small, often less than ten grams. It is necessary to provide enough shielding around the detectors to ensure that the detected radiations come from the waste package, and not the process line. To avoid this problem, a measurement area should be marked off to prevent inadvertent radiation background problems caused by moving plutonium too close to the instruments.

#### 8. Post-Assay Handling

Small packages of waste may be combined in large containers for offsite disposal. Each assayed sealed waste package is weighed and set aside until a sufficient number of packages are accumulated to fill a shipping container. A shipping container is then brought in, examined, and filled. After filling, it is immediately closed and sealed and transferred from the material access area to an approved storage area to await further disposition. Accountability records are prepared by combining the assay values of all the sealed packages loaded into each shipping container.

### C. REGULATORY POSITION

This guide describes procedures for the control and accountability of plutonium-contaminated waste. The procedures are acceptable to the NRC staff for materials protection purposes.

#### 1. Handling Procedures

Plutonium-contaminated waste should be separated into distinct categories, each of which should be packaged separately. Criteria for separation should include density and neutron-moderating considerations, in addition to health and safety criteria.

\*Presently in development. Copies may be obtained from the Institute of Nuclear Materials Management, 505 King Avenue, Columbus, Ohio, 43201. Attention: M. H. L. Toy.

\*\*See Regulatory Guide 5.34, "Nondestructive Assay for Plutonium in Scrap Material by Spontaneous Fission Detection."

## 2. Containers and Packaging Procedures

Waste should be packaged for in-plant handling in small-diameter containers wherever possible. The diameter of the package should be less than 15 cm.\* The same type and size of container should be used for all waste categories except special types of waste (e.g., solutions or large metal pieces). Large items should be disassembled or cut up to facilitate handling, assay, and search procedures. Packages should be heat-sealed in multiple transparent polyethylene bags to avoid contamination when they are removed from approved enclosures. Contaminated tools, machine parts, or sections of vessels or interconnecting plumbing should be thoroughly cleaned to remove as much plutonium as possible before they are packaged as waste.

## 3. Sealing Procedures

Pressure-sensitive seals\*\* should be applied across each heat-sealed closure in the next to the outermost transparent plastic bag. On each package having more than one heat-sealed closure, the identification number of each seal should be written on all other seals on that package. Each completed package should be weighed individually and the weight recorded.

## 4. Assay and Search Procedures

Gamma ray assay procedures should be used whenever the transmission through the entire package from an external source of 414-keV gamma rays is greater than 1%. Packages failing to meet this criterion should be assayed by neutron detection methods. Homogeneous materials containing low concentrations of plutonium may be assayed either by sampling and chemical analysis or by the nondestructive assay procedures described below.

An acceptable upper limit on the plutonium content in each package should be established for each type of waste. Packages indicating higher amounts should be opened and examined for conspicuous attempts to conceal plutonium. The appropriate NRC Regional Office should be notified immediately if a conspicuous attempt to conceal plutonium is detected.

### 4.1 Isotopic Analysis

The isotopic composition of each container of waste should be traceable to a measured value. A previously measured value can be used when verified by measuring the ratio of the intensity of gamma rays from at least

two plutonium isotopes. When the isotopic composition cannot be verified, the package should be either sampled for mass/alpha spectrometric assay (ref. 2) or measured by gamma ray spectral analysis.

## 4.2 Gamma Ray Assay Procedures

Gamma ray assay for plutonium in waste should be accomplished by applying gamma ray spectrometry to each package of plutonium waste by procedures described in reference 1. Of the options discussed in reference 1, those described below are generally acceptable to the NRC staff.

### 4.2.1 Detection System

A high-resolution Ge(Li) gamma ray detection system should be used. The system should have, as a minimum, the performance specifications of a type I system as described in Regulatory Guide 5.9, "Specifications for Ge(Li) Spectroscopy Systems for Material Protection Measurements."

### 4.2.2 Collimation and Scanning Procedures

Each package should be rotated and scanned vertically during assay. The detector should be collimated to view a vertical segment of the container measuring no more than 2-3 cm high. The collimator should be designed to view the entire diameter or width dimension of the package during assay. The intensity of the appropriate gamma rays should be measured independently for each vertical segment. The total package contents should be determined by summing the contributions from all of the contiguous vertical segments.

### 4.2.3 Attenuation Corrections

To measure attenuation corrections, an external source of gamma rays (see ref. 1) should be positioned directly in front of the detector collimator opening, such that the gamma rays emitted by the source will travel through the waste package before striking the detector. The assay results for each segment of each package should be corrected for internal attenuation, based on the measured transmission of the external source gamma rays through that segment. When the transmission at 414 keV through any segment is less than 1%, that package must be searched using the neutron assay procedures described below.

## 4.3 Neutron Assay Procedures

Each waste package that fails to meet the criteria for gamma ray assay should be examined using neutron assay procedures. Regulatory Guide 5.34, "Nondestructive Assay for Plutonium in Scrap Material by Spontaneous Fission Detection," describes procedures that are also applicable to high-density waste. Simple neutron

\*See Regulatory Guide 5.11, "Nondestructive Assay of Special Nuclear Material Contained in Scrap and Waste," for further guidance on this matter.

\*\*See Regulatory Guide 5.10, "Selection and Use of Pressure-Sensitive Seals on Containers for Onsite Storage of Special Nuclear Material."

detection probes may also be applicable.

#### 4.4 Calibration and Error Estimation Procedures

Assay systems should be calibrated and errors determined as described in the publications mentioned below. Guidance related to calibration is described in the LASL report (ref. 1). Error estimation procedures for the separate calculation of random and systematic errors associated with the assay are described in a regulatory guide being prepared, "Calibration and Error Estimation Procedures for Nondestructive Assay." Additional discussion of these topics as they specifically relate to plutonium waste assay is given in the LASL report [ref. 1, Section II, items (a) through (e)].

### 5. Post-Assay Handling

#### 5.1 Interim Storage

Waste packages should be stored until a sufficient quantity is accumulated to fill a shipping container.

#### 5.2 Shipping Container Manifest

A make-up sheet for each shipping container should be prepared. The sheet should list information for each waste package container, including the identifying code of the pressure-sensitive seals, the gross package weight, and the assayed plutonium content. The sheet should identify the types of waste. It should also note if the assay values for concealed plutonium were obtained by sampling and analysis (homogeneous waste only) combined with a neutron search, or by gamma ray assay or neutron assay. The sheet should identify the shipping container and its empty weight, including the weights of all items to be used for closing and sealing.

#### 5.3 Shipping Container Loading and Tamper-Safing Procedures

Immediately prior to the loading of a shipping container, the shipping container should be emptied of all contents and visually examined for integrity and unusual characteristics. A minimum of two operators should perform the loading and sign the make-up sheet. Each container should be continuously observed from the time loading is started until the container is closed and sealed. Each package should be checked for seal and closure integrity as it is loaded into the shipping

container. Care should be exercised to ensure that the containers are not ruptured during loading.

When the shipping container is filled, it should be closed and sealed immediately with tamper-indicating seal(s), each of which bears an identification code (see Regulatory Guide 5.15, "Security Seals for the Protection and Control of Special Nuclear Material"). The identification code(s) of the seal(s) used on each shipping container should be recorded on the container make-up sheet. The sealed shipping container should be promptly transferred from the material access area to an approved onsite storage facility, awaiting offsite shipment.

#### 5.4 Preshipment Verification of Contents

A copy of each shipping container make-up sheet should be maintained in the nuclear material control files. Immediately prior to shipment, each shipping container should be weighed and the weight recorded on the make-up sheet. The integrity and identification of all seals used to seal the shipping container should be checked. The gross weight of each shipping container should be compared with the combined weights of the empty shipping container and the waste packages that have been loaded into that container. When the weights do not match or when the seals are not intact or are not properly identified, the shipping container should be quarantined, opened, and its contents examined.

### D. IMPLEMENTATION

The purpose of this section is to provide information to applicants and licensees regarding the NRC staff's plans for utilizing this regulatory guide.

Except in those cases in which the applicant proposes an alternative method for complying with specified portions of the Commission's regulations, the method described herein will be used in the evaluation of submittals in connection with a special nuclear material license, operating license, or construction permit for applications docketed after October 1, 1975.

If an applicant whose application for a special nuclear material license, an operating license, or a construction permit is docketed on or before October 1, 1975, wishes to use this regulatory guide in developing submittals for applications, the pertinent portions of the application will be evaluated on the basis of this guide.

### REFERENCES

1. T.D. Reilly and J.L. Parker, "A Guide to Gamma Ray Assay for Nuclear Material Accountability," Los Alamos Scientific Laboratory Report LA-5794-MS (1974).
2. See, for example, R. G. Gutmacher, F. Stephens, K. Ernst, J.E. Harrar, J. Magistad, T.E. Shea, and S.P. Turel, "Methods for the Accountability of Plutonium Nitrate Solutions," WASH-1282 (1974).

UNITED STATES  
NUCLEAR REGULATORY COMMISSION  
WASHINGTON, D. C. 20555

OFFICIAL BUSINESS  
PENALTY FOR PRIVATE USE, \$300

POSTAGE AND FEES PAID

UNITED STATES NUCLEAR  
REGULATORY COMMISSION

