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OFFICE OF NUCLEAR REGULATORY RESEARCH

DRAFT REGULATORY GUIDE AND VALUE/IMPACT STATEMENT

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Contact: S. Frattali (301)443-5976

PROPOSED REVISION 1 TO REGULATORY GUIDE 5.34

NONDESTRUCTIVE ASSAY FOR PLUTONIUM IN SCRAP MATERIAL BY SPONTANEOUS FISSION DETECTION

A. INTRODUCTION

Section 70.51, "Material Balance, Inventory, and Records Requirements," of 10 CFR Part 70, "Domestic Licensing of Special Nuclear Material," requires certain licensees authorized to possess at any one time more than one effective kilogram of special nuclear material to establish and maintain a system of control and accountability so that the limit of error associated with the inventory difference (LEID), ascertained as a result of a measured material balance, meets minimum standards. This guide is intended for those licensees who possess plutonium scrap materials and who are also subjected to the requirements of § 70.51 of 10 CFR Part 70. *

Included in a typical material balance are containers of inhomogeneous scrap material that are not amenable to assay by the traditional method of sampling and chemical analysis. With proper controls, the nondestructive assay (NDA) technique of spontaneous fission detection (SFD) is one acceptable method for the assay of plutonium in containers of bulk scrap material. The use of SFD thus facilitates the preparation of a complete plant material balance whose LEID meets established requirements.

This guide describes procedures acceptable to the NRC staff for applying the NDA technique of SFD to plutonium in scrap.

B. DISCUSSION

Plutonium in scrap material can contribute significantly to the inventory difference and its associated limit of error. Unlike the major quantity of

*Lines indicate substantive changes from previous issue.

This regulatory guide and the associated value/impact statement are being issued in draft form to involve the public in the early stages of the development of a regulatory position in this area. They have not received complete staff review and do not represent an official NRC staff position.

Public comments are being solicited on both drafts, the guide (including any implementation schedule) and the value/impact statement. Comments on the value/impact statement should be accompanied by supporting data. Comments on both drafts should be sent to the Secretary of the Commission, U.S. Nuclear Regulatory Commission, Washington, D.C. 20555, Attention: Docketing and Service Branch, by AUG 30 1982

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material flowing through the process, scrap is typically inhomogeneous and difficult to sample. Therefore, a separate assay of the entire content of each container of scrap material is a more reliable method of scrap accountability. NDA is a method for assaying the entire content of every container of scrap.

The term "scrap" refers to material that is generated from the main process stream because of the inefficiency of the process. Scrap material is generally economically recoverable. Scrap, therefore, consists of rejected or contaminated process material such as pellet grinder sludge, sweepings from gloveboxes, dried filter sludge, and rejected powder and pellets. Scrap is generally distinguished from "waste" by the density or concentration of heavy elements in the two materials, but it is the recovery cost (per mass unit of special nuclear material) that determines whether a material is "scrap" or "waste." The concentration of uranium and plutonium in scrap is approximately the same as it is in process material, i.e., 85-90 percent (uranium + plutonium) by weight. However, on occasion the uranium and/or plutonium weight fraction in both process and scrap material can be less than 25 percent. Plutonium in fast reactor scrap material is 15-20 percent by weight and in thermal reactor recycle material, 2-9 percent by weight. The main difference between scrap and process material is that scrap is contaminated and inhomogeneous. Waste, on the other hand, contains a low concentration of uranium and plutonium, i.e., a few percent or less (uranium + plutonium) by weight. However, the recovery of combustible waste by incineration may produce ash that is high in uranium and plutonium concentrations. Such incinerator ash is also considered "scrap" in this guide. However, it should be noted that ash may be more homogeneous in its characteristics compared to most scrap and may, therefore, be accountable using sampling and chemical analysis methods.

NDA of plutonium can be accomplished primarily by the passive methods of gamma ray spectrometry, calorimetry, and SFD. Active neutron methods using total count rates and/or delayed neutron detection can also be used in scrap assay measurements. Regulatory Guide 5.11, "Nondestructive Assay of Special Nuclear Material Contained in Scrap and Waste," provides a framework for the use of these NDA methods.

Gamma ray spectrometry of scrap consisting of dense materials can be unreliable because of the attenuation of gamma rays. However, the isotopic

composition of plutonium in scrap materials can be obtained quite reliably using high-resolution gamma ray spectrometry measurements (Ref. 1).

Calorimetry is an accurate method of plutonium assay when there is an accurate knowledge of the relative abundance of each plutonium isotope and americium-241. Scrap may contain a mixture of materials of different radionuclidic compositions, especially different americium-241 concentrations, thereby necessitating the measurement of the average radionuclidic composition. The average radionuclidic abundances can only be accurately measured when the scrap is reasonably homogeneous. When the radionuclidic abundances can be accurately measured or controlled, calorimetry can be applied to scrap assay (Ref. 2). However, calorimetry is time-consuming for materials of high heat capacity and may not be a practical method for the routine assay of large numbers of containers.

SFD is a practical NDA technique for the assay of plutonium in scrap material. The assay method involves the passive counting of spontaneous fission neutrons emitted primarily from the fission of plutonium-240. Neutron coincidence counters are used to detect these time-correlated neutrons. The theory and practice of neutron coincidence counting for plutonium assay are discussed thoroughly in References 3-5. Spontaneous fission neutrons are sufficiently penetrating to provide a representative signal from all the plutonium within a container. Since the neutron coincidence signal is dependent upon both the quantity and relative abundance of plutonium-238, plutonium-240, and plutonium-242, the plutonium isotopic composition must be known for SFD assay of total plutonium. However, the accuracy of SFD is not as dependent upon the accuracy of analysis for the minor plutonium isotopes as is that of calorimetry. Nor is SFD sensitive to americium-241 ingrowth. The quantity of scrap material on inventory when a material balance is computed can be reduced through good management, and the scrap remaining on inventory can be assayed by SFD to meet the overall plant inventory difference (ID) and LEID constraints required by paragraph 70.51(e)(5) of 10 CFR Part 70.

This guide gives recommendations useful for the SFD assay of containers, each containing a few liters of scrap and having contents ranging from a few grams to 10 kilograms of plutonium or up to approximately 2 kilograms of

effective plutonium-240* (see Ref. 6). Containers with a significant plutonium content (i.e., on the order of 50 grams or more) give a spontaneous fission response that must be corrected for the effects of neutron multiplication (Refs. 7 and 8). Scrap materials that have large loadings of plutonium in addition to fluorine, oxygen, or other alpha/neutron-producing elements are difficult to measure and correct for multiplication effects because of the large random neutron flux from the (α,n) reactions in the matrix materials. These samples should be segregated into smaller quantities for measurements. In general, a large quantity of plutonium can be assayed by SFD by subdividing the scrap into smaller amounts, or the items may be more amenable to assay by calorimetry.

C. REGULATORY POSITION

The SFD method for the NDA of plutonium in bulk inhomogeneous scrap material should include (1) discrimination of spontaneous fission radiations from random background by coincidence techniques and (2) measurement of the relative plutonium isotopic composition of the scrap. An acceptable SFD method of plutonium assay is described below.

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* The effective plutonium-240 mass is a weighted average of the mass of each of the plutonium isotopes. The weighting is equal to the spontaneous fission neutron yield of each isotope relative to that of plutonium-240. Since only the even-numbered isotopes have significant spontaneous fission rates, the effective plutonium-240 mass is given approximately by:

$$M(240)_{eff} = M(240) + 1.64M(242) + 2.66M(238)$$

where M is the mass of the isotope indicated in parentheses. The coefficients in this equation are known only to approximately ±5 percent.

Another form of the equation frequently used is:

$$M(240)_{eff} = M(240) + 1.57M(242) + 2.43M(238).$$

The mathematical procedure for conversion from $M(240)_{eff}$ to M(total Pu) is described in the appendix to this guide, together with a sample calculation.

1. SPONTANEOUS FISSION DETECTION SYSTEM

1.1 Detectors

Instruments based on moderated thermal neutron detectors, i.e., neutron well coincidence counters (Refs. 4, 5, and 9), are recommended for applications in which the gross neutron detection rate does not exceed 2×10^5 neutrons/sec. The dead time inherent in these slow coincidence systems can be reduced by employing a shift-register coincidence circuit (Refs. 10 and 11). If the gross neutron detection rate is primarily due to random background and exceeds 2×10^5 neutrons/sec, then a fast-neutron-detection, single-coincidence system can be used, provided adequate corrections can be made for matrix effects. Matrix effects are more severe in fast-neutron-detection systems, as shown in Table 1.

1.2 Detection Chamber

The chamber should permit reproducible positioning of standard-sized containers in the location of maximum spatial response uniformity.

1.3 Fission Source

A spontaneous fission source with a neutron intensity comparable to the intensity of the largest plutonium mass to be assayed should be used for making matrix corrections, using the source addition technique (Ref. 12). A nanogram of californium-252 is approximately equivalent to a gram of effective plutonium-240.

1.4 Readout

Readout should allow computation of the accidental-to-real-coincidence ratio in addition to the net real-coincidence rate. Live-time readout or a means of computing the dead time should also be provided.

TABLE 1

MATRIX MATERIAL EFFECTS ON NEUTRON ASSAY

Matrix Material (in ~4-liter can)	Mass (kg)	Neutron Detection Efficiency (Ref. 13)			Coincidence Efficiency, He-3 Detector, Thermal	Corrected[a] (Ref. 12) Coincidence Efficiency He-3 Detector, Thermal
		He-3 Detector, Thermal	He-4 Detector, Fast	ZnS Detector, Fast		
Empty Can	--	1.00	1.00	1.00	1.00	1.00
Carbon Pellets	1.89	1.03	--	--	1.05	0.97
Metal	3.60	1.04	0.83	0.75	1.09	1.02
Slag-Crucible	1.80	1.03	0.94	0.91	1.08	1.01
Concrete	3.24	1.05	0.84	0.79	1.10	1.02
String Filters	0.60	1.07	0.95	0.86	1.17	1.05
CH_2 (ρ=0.65 g/cc)	0.27	1.06	0.96	0.92	1.11	1.00
CH_2 (ρ=0.12 g/cc)	0.43	1.09	0.92	0.90	1.19	0.98
CH_2 (ρ=0.27 g/cc)	0.97	1.19	0.71	0.67	1.36	0.04
H_2O (ρ=1.00 g/cc)	3.62	0.98	0.36	0.35	0.98	0.96

[a]Corrected using the source addition technique (see Ref. 6).

1.5 Performance Specifications

The performance of an SFD instrument should be evaluated according to its stability, uniformity of spatial response, and insensitivity to matrix effects. Therefore, information should be obtained regarding:

a. The precision of the coincidence response as a function of the real-coincidence counting rate and the accidental-to-real-coincidence ratio. Extremes in the background or accidental-coincidence rate can be simulated by using a source of random neutrons (nonfission).

b. Uniformity of spatial response. Graphs should be obtained on the relative coincidence response to a small fission neutron source as a function of position in the counting chamber.

c. Sensitivity of matrix interference. A table of the relative coincidence response to a small fission neutron source as a function of the composition of the matrix material surrounding the point source should be obtained. Included in the matrix should be materials considered representative of common scrap materials. Table 1 is an example of such a tabulation of the relative response for a wide range of materials.

This information should be used for evaluating the expected instrument performance and for estimating errors. The above performance information can be requested from the instrument suppliers during instrument selection and should be verified during preoperational instrument testing.

2. ANALYST

A trained individual should oversee SFD assay of plutonium and should have primary responsibility for instrument specification, preoperational instrument testing, standards and calibration, an operation manual, measurement control, and error analysis. Experience or training equivalent to a bachelor's degree in science or engineering from an accredited college or university and a laboratory course in radiation measurement should be the minimum qualifications of the SFD analyst. The SFD analyst should frequently review SFD operation and should authorize any changes in the operation.

3. CONTAINERS AND PACKAGING

A single type of container should be used for packaging all scrap in each category. A uniform container that would facilitate accurate measurement and would standardize this segment of instrument design such as a thin-walled metal (steel) can with an inside diameter between 10 and 35 cm is recommended. For further guidance on container standardization in NDA measurements, see Reference 14.

4. REDUCING ERROR DUE TO MATERIAL VARIABILITY

The SFD response variation due to material variability in scrap should be reduced by (1) segregating scrap into categories that are independently calibrated, (2) correcting for matrix effects using the source addition technique (Ref. 12), or (3) applying both the categorization and the source addition technique. Categorization should be used if the SFD method is more sensitive to the material variability from scrap type to scrap type than to the material variability within a scrap type. Application of the source addition technique reduces the sensitivity to material variability and may allow the majority of scrap types to be assayed under a single calibration. Material characteristics that should be considered in selecting categories include:

a. Plutonium isotopic composition and content,

b. Uranium/plutonium ratio,

c. Types of container and packaging,

d. Abundance of high-yield alpha-neutron material, i.e., low-atomic-number impurities,

e. Size and distribution of materials in packages,

f. Density (both average density and local density extremes should be considered), and

g. Matrix composition.

5. CALIBRATION

Guidelines for calibration and measurement control for NDA are available in Regulatory Guide 5.53, "Qualification, Calibration, and Error Estimation

Methods for Nondestructive Assay," which endorses ANSI N15.20-1975.* A proposed revision to this guide is currently being developed. The guide and standard include details on calibration standards, calibration procedures, curve fitting, and error analysis. Guidelines relevant to SFD are given below.

Calibration can be used for either a single isotopic composition or variable isotopic mixtures. In the former case, the resulting calibration curve will be used to convert "net real-coincidence count" to "grams plutonium." In the latter case, the conversion is from "net real-coincidence count" to "effective grams plutonium-240." The mathematical procedure for conversion from effective grams plutonium-240, $M(240)_{eff}$, to total grams plutonium, M(total Pu), is described in the appendix to this guide along with a sample calculation.

A minimum of four calibration standards of similar isotopic composition to the unknowns should be used for calibration. If practicable, a calibration curve should be generated for each isotopic blend of plutonium. When plutonium of different isotopic composition is assayed using a single calibration, the effect on the SFD response of isotopic composition should be determined over the operating ranges by measuring standards of differing plutonium isotopic compositions. This is necessary since the use of the effective plutonium-240 concept can lead to error because of the uncertainty in the spontaneous fission half-lives, as shown in Table 2 (Ref. 13), and the variation in response with isotopic composition.

Calibration standards should be fabricated from material having a plutonium content determined by a technique traceable to or calibrated with the standard reference material at the National Bureau of Standards. Well-characterized homogeneous material similar to the process material from which the scrap is generated can be used to obtain calibration standards.

Fabrication of calibration standards that are truly representative of the unknowns is impossible for scrap assay. To measure the reliability of the calibration based on the fabricated standards discussed above and to improve this calibration, unknowns that have been assayed by SFD should periodically be selected for assay by an independent technique. Calorimetry (Ref. 2) can be used to assay a random selection of scrap in containers and to provide

*Copies of this standard may be obtained from the American National Standards Institute, Inc., 1430 Broadway, New York, New York 10018.

TABLE 2

EFFECTIVE Pu-240 ABUNDANCE AND UNCERTAINTY CORRESPONDING TO DIFFERENT ISOTOPIC COMPOSITION[a,b]

BURNUP (MWd/t)	Approximate Abundance (%)				
	Pu-239	Pu-240	Pu-241	Pu-242	$Pu\text{-}240_{eff}$
8,000-10,000	87	10	2.5	0.3	10.75±0.03(0.3%)
16,000-18,000	75	18	4.5	1.0	20.30±0.08(0.4%)
25,000-27,000	58	25	9.0	7.0	39.14±0.50(1.3%)
38,000-40,000	45	27	15.0	12.0	52.00±0.87(1.7%)

[a]Computed using the following coefficients for plutonium-238 and plutonium-242 in the equation for plutonium-240 effective:

$$M(240)_{eff} = M(240) + 1.64 \pm 0.07M(242) + 2.66 \pm 0.19M(238)$$

The uncertainties in the coefficients and in the effective plutonium-240 abundances in the table are from the reported standard deviations in the most reliable data available (Ref. 6).

[b]Plutonium isotopic compositions were selected based on light-water-reactor fuel exposures.

reliable data that should be fed back into the calibration fitting procedure to improve SFD calibration. The original calibration standards should be retained as working standards.

6. MEASUREMENT CONTROL

For proper measurement control, on each day that scrap is assayed, a secondary standard should be assayed as a background measurement. Also, on each day that scrap is assayed, control (or working) standards should be assayed for normalization and for ensuring reliable operation.

The source addition technique (Ref. 12) is recommended for correcting the SFD response for each assay. If not used routinely, the source addition technique should be applied to a random selection of items with a frequency comparable to the assay schedule. The results of random applications of the source addition technique can be used in two ways:

a. As an average correction factor to be applied to a group of items, and

b. As a check on the item being assayed to verify that it is similar to the standards used in calibration and that no additional matrix effects are present, i.e., purely as a qualitative assurance that the calibration is valid.

7. ERROR ANALYSIS

The sources of error in SFD are discussed in Regulatory Guide 5.11. Analysis of the error in the calibration is discussed in ANSI N15.20-1975 and in References 4 and 15.

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APPENDIX

Procedure for Conversion of $M(240)_{eff}$ to M(total Pu) and Sample Calculation

When the measurement situation dictates the expression of the primary assay result as "effective grams plutonium-240," it is necessary to convert this result to total grams plutonium using the relationship between these two quantities and the known isotopic composition of the plutonium sample. Let f_{238}, f_{239}, f_{240}, f_{241}, f_{242} represent the weight fractions of the respective plutonium isotopes in the unknown sample. The effective plutonium-240 mass from coincidence counting, $M(240)_{eff}$, and the individual masses of the spontaneously fissioning plutonium isotopes are related by:

$$M(240)_{eff} = M(240) + 1.64M(242) + 2.66M(238). \quad (1)$$

The masses of the plutonium-242 and plutonium-238 isotopes can be expressed in terms of M(240), using the isotopic weight fractions, so that:

$$M(240)_{eff} = M(240)[f_{240} + 1.64f_{242} + 2.66f_{238}]/f_{240}. \quad (2)$$

Since $M(240)/f_{240} = M(\text{total Pu})$, we have the final results:

$$M(\text{total Pu}) = M(240)_{eff}/[f_{240} + 1.64f_{242} + 2.66f_{238}]. \quad (3)$$

The quantity in the denominator of Equation (3) is called the "plutonium-240 effective weight fraction, f_{240}(effective)." Thus, the total plutonium mass can be expressed as the plutonium-240 effective mass divided by the plutonium-240 effective weight fraction:

$$M(\text{total Pu}) = M(240)_{eff}/f_{240}(\text{effective}). \quad (4)$$

As an example, suppose that the net coincidence count from an unknown sample indicates 10.0 ± 0.5 effective grams of plutonium-240. Furthermore, suppose that the plutonium isotopic composition of the unknown sample was previously established to be:

f_{238} = (1.0 ± 0.5) % (= 0.010 ± 0.005)
f_{239} = (73.0 ± 0.5)%
f_{240} = (20.0 ± 0.4)% (= 0.200 ± 0.004)
f_{241} = (4.0 ± 0.2) %
f_{242} = (2.0 ± 0.2) % (= 0.020 ± 0.002).

Using these results in Equation (3), we have:

M(total Pu) = 10.0/[0.20 + 1.64x0.02 + 2.66x0.01]
= 10.0/0.259 = 38.6 grams.

To obtain the value of the variance of the M(total Pu) result, we must propagate the quoted variances of the $M(240)_{eff}$ and the isotopic weight fractions. Let the variance in $M(240)_{eff} = \sigma_{eff}$, and let the variances in the relevant plutonium weight fractions be σ_{238}, σ_{240}, and σ_{242}. The variance of the total plutonium mass, σ_{Pu}, is given by:

$$[\sigma_{Pu}/M(\text{total Pu})]^2 = [\sigma_{eff}/M(240)_{eff}]^2$$

$$+ [\sigma_{240}^2 + (1.64\sigma_{242})^2 + (2.66\sigma_{238})^2]/[f_{240} + 1.64f_{242} + 2.66f_{238}]^2. \quad (5)$$

In our example calculation, σ_{eff} = 0.5 gram, σ_{238} = 0.005, σ_{240} = 0.004, and σ_{242} = 0.002. The variance in the total plutonium mass is therefore given by:

$$\sigma_{Pu} = M(\text{total Pu})\,[(0.5/10.0)^2 + 0.000204/(0.259)^2]^{\frac{1}{2}}$$
$$= 38.6 \times 0.074 = 2.9 \text{ grams.}$$

Thus, the final assay result from this coincidence count is quoted as:

M(total Pu) = 38.6 ± 2.9 grams.

For most plutonium samples, the dominant measurement uncertainties will be in the plutonium-240 effective mass and the plutonium-240 isotopic weight fraction, f_{240}. Thus, good precision in M(total Pu) is achieved primarily through minimizing the uncertainties in these quantities.

DRAFT VALUE/IMPACT STATEMENT

1. PROPOSED ACTION

1.1 Description

Licensees authorized to possess at any one time more than one effective kilogram of plutonium are required in § 70.51 of 10 CFR Part 70, "Domestic Licensing of Special Nuclear Material," to establish and maintain a system of control and accountability so that the limit of error associated with the inventory difference (LEID), ascertained as a result of a measured material balance, meets minimum standards.

Included in a typical material balance are containers of inhomogeneous scrap material that are not amenable to assay by the traditional method of sampling and chemical analysis. With proper controls, the nondestructive assay (NDA) technique of spontaneous fission detection (SFD) is one acceptable method for the assay of plutonium in containers of bulk scrap material. The use of SFD thus facilitates the preparation of a complete plant material balance whose LEID meets established requirements.

This guide describes procedures acceptable to the NRC staff for applying the NDA technique of SFD to plutonium in scrap.

1.2 Need for Proposed Action

The proposed action is needed to bring Regulatory Guide 5.34 up to date.

1.3 Value/Impact of Proposed Action

1.3.1 NRC Operations

The experience and improvements in technology that have occurred since the guide was issued will be made available for the regulatory procedure. 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 the technique and to bring the language of the guide into conformity with current usage.

2. TECHNICAL APPROACH

Not applicable.

3. PROCEDURAL APPROACH

3.1 Procedural Alternatives

Potential RES procedures that may be used to promulgate the proposed action and technical approach include the following:

- Regulation
- Revision of regulatory guide
- ANSI standard, endorsed by regulatory guide
- Branch position
- NUREG report

3.2 Discussion of Procedural Alternatives

Since a useful and usable regulatory guide exists and modifications are minimal, the simplest procedure is to revise the guide.

3.3 Decision on Procedural Approach

A revised regulatory guide should be prepared.

4. STATUTORY CONSIDERATIONS

4.1 NRC Authority

Authority for this guide would be derived from the safety requirements of the Atomic Energy Act through the Commission's regulations, in particular, § 70.51 of 10 CFR Part 70.

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 the first of a series of revisions of existing regulatory guides on nondestructive assay techniques.

6. SUMMARY AND CONCLUSIONS

Regulatory Guide 5.34 should be updated.

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

119406002001 1 QPSAS5
US NRC REGION I
R J BORES
631 PARK AVENUE
REGION I
KING OF PRUSSIA PA 19406