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**Gamma-Ray Isotopic Ratio
Measurements for the Plutonium
Inventory Verification Program**

*John F. Lemming, Francis X. Haas
and Jack Y. Jarvis*

August 25, 1976

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ABSTRACT

The Plutonium Inventory Verification Program at Mound Laboratory provides a nondestructive means of assaying bulk plutonium-bearing material. The assay is performed by combining the calorimetrically determined heat output of the sample and the relative abundances of the heat-producing isotopes. This report describes the method used for the nondestructive determination of plutonium-238, -240, -241 and americium-241 relative to plutonium-239 using gamma-ray spectroscopy for 93% plutonium-239 material. Comparison of chemical data on aliquots of samples to the nondestructive data shows accuracies of $\pm 7\%$ for $^{239}\text{Pu}/^{239}\text{Pu}$, $\pm 15\%$ for $^{240}\text{Pu}/^{239}\text{Pu}$, $\pm 3\%$ for $^{241}\text{Pu}/^{239}\text{Pu}$, and $\pm 7\%$ for $^{241}\text{Am}/^{239}\text{Pu}$.

INTRODUCTION

The Plutonium Inventory Verification Program¹⁻⁴ was established at Mound Laboratory to give an independent nondestructive verification of assay measurements at contractor facilities.

As a part of this program, auditors select samples from contractor inventories and send them to Mound Laboratory for calorimetric assay.⁵ The variety of categories which have been assayed are oxide, metal, incinerator ash, ash heel, plutonium-238 scrap, fluoride, crucibles, slag, scarfings, and green cake.

The scrap samples are normally packaged in gallon cans containing 20 to 400 g of

plutonium. The remaining samples are packaged in quart cans containing 10 to 2500 g of plutonium.

This report describes the gamma-ray spectroscopic techniques used to nondestructively determine the isotopic ratios necessary for calorimetric assay. Time limitations on the inventory require three samples to be processed per day, limiting gamma-ray data acquisition times to approximately 4 hr. The data contained in this report were obtained in the time phase ending December 31, 1975.

TECHNIQUE

The bases for the nondestructive measurement of plutonium isotopic ratios using passive high-resolution gamma-ray spectrometers have been discussed and reviewed by several authors.⁶⁻¹⁵ In general, a spectral group whose members belong to different isotopes is analyzed. Ratios of the areas of gamma-ray peaks are related to the isotopic abundance ratios by the expression:

$$\frac{I_i}{I_j} = \frac{N_i \lambda_i B_i \epsilon_i S_i}{N_j \lambda_j B_j \epsilon_j S_j}$$

where I , N , λ , B , ϵ and S are the measured peak area, the number of nuclei, the nuclear decay constant, the absolute branching intensity, the detector efficiency, and the self-absorption for the gamma-rays with energies E_i and E_j from isotopes i and j , respectively.

The relative efficiency, which is a product of the detector efficiency and the absorption coefficient, varies with energy. Although the efficiency curve can be measured, the absorption effects from an unknown package are often impossible to predict. For this reason, gamma-ray peak pairs whose energies differ by less than 10 keV are chosen, and the assumption is made that the ratio of the relative efficiencies is 1.

From a study of the published decay data on the plutonium isotopes and americium-241, several sets of peak pairs were

chosen for calculating isotopic ratios. Table 1 is a summary of these gamma-ray energies, the branching ratios, and the half-lives necessary for the calculations. The nuclear decay constants have been taken from Gunnink and Morrow.¹⁶

Table 1

LIST OF GAMMA-RAYS AND BRANCHING INTENSITIES OF ISOTOPES OF INTEREST

Isotope	$T_{1/2}$ (days)	Energy (keV)	Branching Intensity
^{238}Pu	3.2×10^4	152.77	1.01×10^{-5}
^{239}Pu	8.908×10^6	141.64	3.11×10^{-7}
		144.19	2.84×10^{-6}
		146.05	1.13×10^{-6}
		203.52	5.63×10^{-6}
		637.97	2.50×10^{-8}
		640.15	7.95×10^{-8}
		658.99	9.50×10^{-8}
^{240}Pu	2.41×10^6	642.30	1.45×10^{-7}
^{241}Pu	2.070×10^8 ^a	148.60	7.74×10^{-2}
^{237}U	6.75	207.97	2.080×10^{-1}
^{241}Am	1.582×10^5	146.55	4.58×10^{-6}
		207.98	7.60×10^{-6}
		662.37	3.46×10^{-6}

^aAlpha half-life.

DATA ACQUISITION

The accuracy and reproducibility of the isotopic ratios determined by gamma-ray spectroscopy depend on the reliability of the extracted gamma-ray peak areas. The primary contribution to the uncertainties associated with each peak area is a function of the resolution of the gamma-ray spectrometer. Hence, it is recommended that an optimum choice be made of the detector and the electronics to be used (e.g., see Regulatory Guide 5.9).¹⁷

The detector system used for our program is a 70-cc Ge(Li) crystal with a resolution of 1.80 keV for the 1332-keV cobalt-60 gamma-ray. The gain on the linear amplifier is adjusted so that the energy calibration is 0.26 keV/channel. A 4096-channel analyzer is used to acquire the data. A 0.030-in. (0.076-cm) cadmium absorber is used to suppress low-energy gamma-rays. The sample-to-detector distance is chosen to give a count rate between 2500 and 4000 counts/sec. Samples are counted for approximately 4 hr. The isotopic composition of the plutonium

material is assumed to be homogeneous. However, the sample is rotated during counting to average inhomogeneities due to distribution of the plutonium in the sample. Figure 1 shows a spectrum of an oxide sample acquired using the inventory spectrometer described. The peaks which are used in determining the isotopic ratios are labeled.

In addition to the bulk samples, aliquots (1-5 g of material depending on the category) are chosen from selected samples before shipping to Mound Laboratory. These aliquots are assayed using gamma-ray spectroscopy and then dissolved for analysis by radiocounting and mass spectroscopy to aid in the construction of the isotopics and to establish further reliability upon the gamma-ray techniques. The aliquots are packaged in 5-dram glass vials inside aluminum secondary calorimeter cans (1-1/8 in. [2.86 cm] x 2-9/16 in. [6.51 cm]). Because of their lower count rates the aliquots are counted for 8 hr.

DATA ANALYSIS

The spectra are analyzed on an IBM-360/50 computer using the program GAUSS V.¹⁸ This program uses a nonlinear least-squares fitting technique to describe the data as Gaussian-shaped peaks on a linear background. The program can be used to extract peak areas for a maximum of five overlapping gamma-ray peaks.

The gamma-ray peak areas are converted to isotopic ratios by the computer program ISOTOP. The branching ratios of Gunnink

and Morrow¹⁶ are used since they give the best agreement with chemical analysis. Table 2 summarizes the conversion factors.

In order to verify the operation of the gamma-ray spectrometer, a National Bureau of Standards reference source (NBS-948) whose plutonium characteristics are similar to those of the samples to be inventoried is measured before and after each set of samples.

Table 2

GAMMA-RAYS AND CONVERSION FACTORS USED FOR ISOTOPIC DETERMINATIONS

<u>Isotopic Ratio</u>	<u>Gamma-Rays Used</u>	<u>Conversion Factors to Express Ratio in ppm</u>
$^{238}\text{Pu}/^{239}\text{Pu}$	153 keV/144 keV	$1010.3 \frac{I_{153}}{I_{144}}$
$^{240}\text{Pu}/^{239}\text{Pu}$	642 keV/640 keV	$148330 \frac{I_{642}}{I_{640}}$
$^{241}\text{Pu}/^{239}\text{Pu}$	208 keV/203 keV	$623.7 \left\{ \frac{I_{208}}{I_{203}} - 0.0373 \frac{I_{662}}{I_{659}} \right\}$
	148 keV/144 keV	$852.7 \frac{I_{148}}{I_{144}}$
$^{241}\text{Am}/^{239}\text{Pu}$	662 keV/659 keV	$487.5 \frac{I_{662}}{I_{659}}$

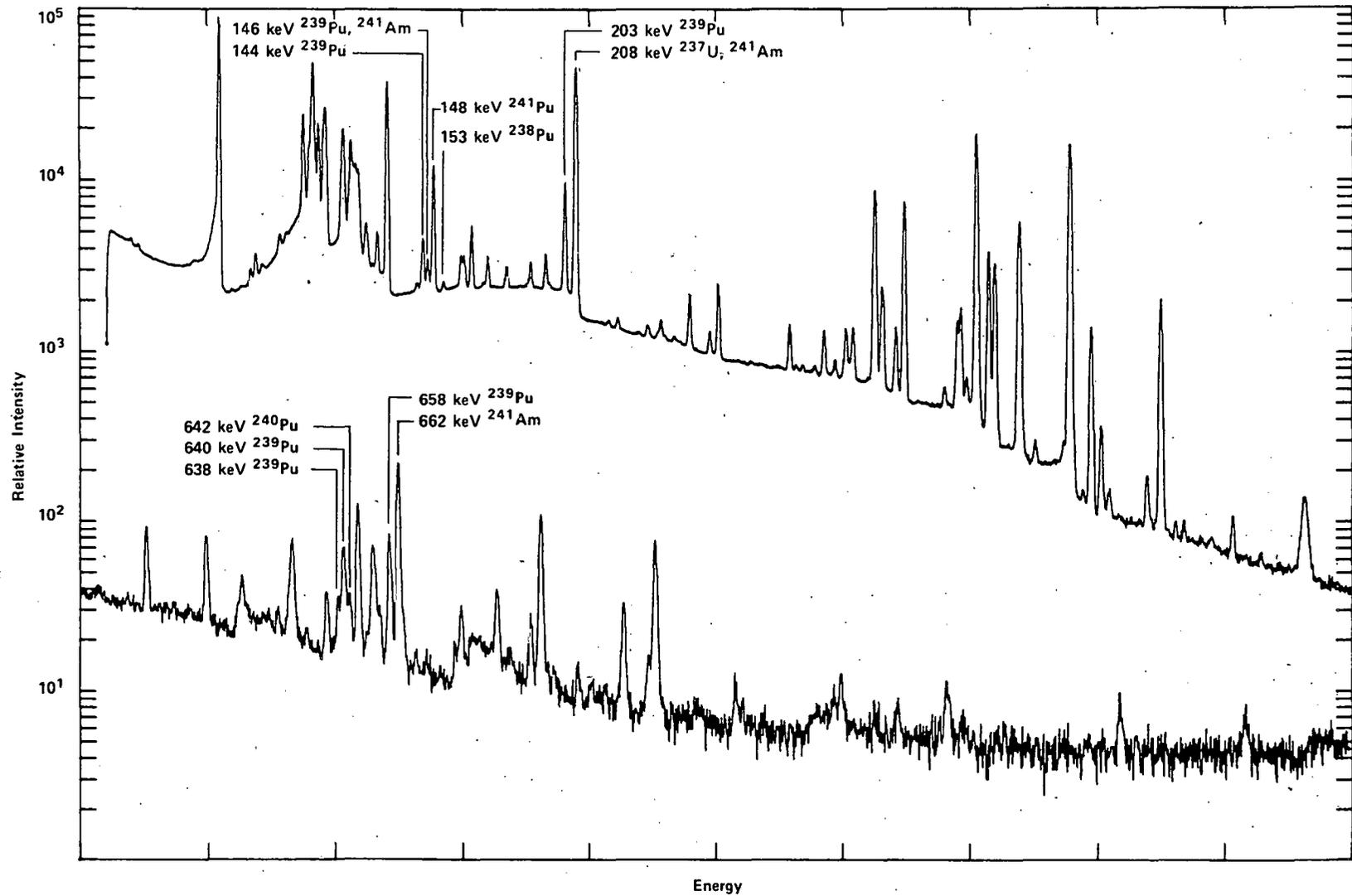


FIGURE 1 - Spectrum of a 292-g oxide sample from the Plutonium Inventory Verification Program obtained with 70-cc Ge(Li) detector No. 813.

RESULTS

Because each set of peak pairs used for the isotopic ratio determinations has its own advantages and disadvantages, the ratios will be discussed individually. It should also be reemphasized that the spectra are acquired for approximately 4 hr and that all isotopic ratios are determined from the same set of data. If only one isotopic ratio were necessary or if a multidetector system were used, the experiments could be tailored to improve the accuracies of the individual measurements.

In the following discussions, the isotopic ratios determined by the gamma-ray technique on the aliquots are compared to those determined by radiocounting and/or mass spectroscopy where appropriate. The isotopic ratios measured on the bulk samples are compared to those measured from the aliquot. All of these samples contain approximately 93% plutonium-239.

$^{241}\text{Pu}/^{239}\text{Pu}$ Ratio

The $^{241}\text{Pu}/^{239}\text{Pu}$ ratio is measured using two sets of peak pairs. The 203.5-keV gamma-ray from plutonium-239 and the 208.0-keV gamma-ray from the uranium-237 daughter of plutonium-241 are a convenient pair when uranium-237 is in equilibrium with the parent plutonium-241. The advantage of this peak pair is that it can be used over a wide range of isotopic fractions. The disadvantage is that a correction must be made for the americium-241 contribution to the 208-keV peak. The other set of peaks which is suitable for most of the inventory material is the 148.6-keV gamma-ray of plutonium-241 and the 144.2-keV gamma-ray of plutonium-239. This peak pair cannot be used for reactor-grade plutonium since the area of the 144.2-keV gamma-ray peak cannot be determined with sufficient precision due to its low branching intensity and uranium interferences. Table 3 is a summary of

the results from four Plutonium Inventory Series. In this table, the value obtained from the 140-keV region is compared to that obtained from the 200-keV region. The weighted average from these two values is used for the comparison to mass spectroscopy data and for the comparison of the bulk samples to their aliquots.

Precision runs to check the reproducibility on selected samples have a relative standard deviation of $\pm 1.1\%$. In order to be conservative the uncertainty used for the inventory program is $\pm 3\%$.

$^{238}\text{Pu}/^{239}\text{Pu}$ Ratio

The 152.8-keV gamma-ray of plutonium-238 is used with the 144.2-keV gamma-ray of plutonium-239 in those cases where the $^{241}\text{Pu}/^{239}\text{Pu}$ ratio determined by the 148.6-keV/144.2-keV peak pair agrees with that determined by the 208.0-keV/203.5-keV peak pair. If the 144.2-keV gamma-ray cannot be used, the $^{238}\text{Pu}/^{241}\text{Pu}$ ratio is calculated using the 152.8-keV gamma-ray of plutonium-238 and the 148.6-keV gamma-ray from plutonium-241. The conversion to the $^{238}\text{Pu}/^{239}\text{Pu}$ ratio is made by using the $^{241}\text{Pu}/^{239}\text{Pu}$ ratio determined from the 208.0-keV/203.5-keV peak pair.

Table 4 is a comparison of the radiocounting ratio to that obtained by gamma-ray techniques for aliquot samples. Except for the Spring '75 series, the agreement with the radiocounting data is within the $\pm 6.5\%$ expected from this peak pair.¹⁹ For the Spring series, some ambiguities exist in the choice of background for the 152.8-keV peak in the running of GAUSS V. Up to 15% differences in peak area were noted for equally valid input parameters. This problem exists for only a few samples. When the 153-keV peak was investigated with a 1-cc LEPS detector (resolution of 600 eV for the 122-keV peak of cobalt-57), two peaks were

Table 3

Inventory Series	COMPARISON OF $^{241}\text{Pu}/^{239}\text{Pu}$ RATIOS		
	$\frac{148/144}{208/203}$	Gamma Aliquot/ Mass Spec	Gamma Can/ Gamma Aliquot
Summer '74	-- ^a	0.97 \pm 0.05	1.03 \pm 0.08
Winter '74	1.02 \pm 0.03	0.96 \pm 0.02	0.98 \pm 0.05
Spring '75	1.03 \pm 0.03	1.01 \pm 0.02	1.01 \pm 0.01
Summer '75	1.01 \pm 0.01	0.98 \pm 0.03	1.01 \pm 0.02

^aFor the Summer '74 series only the 208-keV/203-keV peak pair was used.

resolved in this region. The origin of the background peak remains unsolved. Americium-241 has been ruled out since the concentrations vary from 200 to 3700 ppm. In all cases when using gamma-ray spectroscopy for isotopic determinations, care must be taken that background gamma-rays which contribute to the regions of interest do not go undetected.

For bulk samples the background problems in the 152.8-keV region are more pronounced and the uncertainties are generally near 15%.

Table 4

COMPARISON OF $^{238}\text{Pu}/^{239}\text{Pu}$ RATIOS

Inventory Series	Gamma Aliquot/ Radiocounting	Gamma Can/Gamma Aliquot
Summer '74	0.93 ± 0.06	1.04 ± 0.20
Winter '74	1.00 ± 0.05	1.07 ± 0.16
Spring '75	1.11 ± 0.14	1.43 ± 0.27
Summer '75	0.99 ± 0.07	1.00 ± 0.14

$^{240}\text{Pu}/^{239}\text{Pu}$ Ratio

This ratio is the most difficult one to determine. The method used employs the 642.3-keV gamma-ray from plutonium-240 and the 646.0-keV gamma-ray from plutonium-239. There are several americium-241 gamma-ray peaks in this area which complicate the spectrum analysis. This pair of gamma-rays has very low intensities and the approximately 4-hr counting times are not long enough to measure the ratio to closer than 15%. Table 5 shows the comparison of the mass spectroscopic value to that determined by gamma-ray spectroscopy. With better statistics, this peak pair can be used to give the $^{240}\text{Pu}/^{239}\text{Pu}$ ratio to 1%.^{19,20}

DISCUSSION

The results of the Plutonium Inventory Verification Program, to date, have demonstrated the usefulness of nondestructive gamma-ray isotopic ratio measurements for 93% plutonium-239 samples. For bulk samples the isotopic ratios necessary for calorimetric assay can be determined in a 4-hr counting period to the following accuracies: $^{238}\text{Pu}/^{239}\text{Pu}$ ±15%; $^{240}\text{Pu}/^{239}\text{Pu}$ ±15%; $^{241}\text{Pu}/^{239}\text{Pu}$ ±3%; and $^{241}\text{Am}/^{239}\text{Pu}$ ±7%. When these ratios are combined the resulting uncertainty in the ratio of

Table 5

COMPARISON OF $^{240}\text{Pu}/^{239}\text{Pu}$ RATIOS

Inventory Series	Gamma Aliquot/ Mass Spec	Gamma Can/Gamma Aliquot
Summer '74	0.88 ± 0.14	1.05 ± 0.15
Winter '74	0.90 ± 0.06	0.92 ± 0.09
Spring '75	0.94 ± 0.11	1.03 ± 0.11
Summer '75	0.93 ± 0.11	1.01 ± 0.18

$^{241}\text{Am}/^{239}\text{Pu}$ Ratio

The 662-keV/659-keV peak pair is used to obtain the $^{241}\text{Am}/^{239}\text{Pu}$ ratio. This region assumes that there is no cesium-137 in the source. Table 6 shows a comparison of bulk sample ratios to those determined from their aliquots. The uncertainty assigned to this ratio for the inventory program is ±7%. Precision runs to check the reproducibility yield ±3.3%.

Table 6

COMPARISON OF $^{241}\text{Am}/^{239}\text{Pu}$ RATIOS

Inventory Series	Gamma Can/Gamma Aliquot
Summer '74	1.03 ± 0.18
Winter '74	0.95 ± 0.13
Spring '75	1.01 ± 0.07
Summer '75	1.01 ± 0.07

plutonium-239 to total plutonium is 1%. The isotopic ratio uncertainties result in an uncertainty of 3% in converting a 1-W sample to grams of plutonium. (The calorimeter's thermal power error has been assumed to be 0.15%.) Table 7 shows the results from the computer program CALDAT which uses the formulae of ANSI Standard 15.22²¹ to determine the weight of plutonium in grams and its uncertainty.

Table 7

SAMPLE PRINTOUT FROM CALDAT

4 FEB 1976

SAMPLE ID = 1 WATT
DATE CALORIMETERED 2/4/76
238-PU ATOM RATIO = 115
240-PU ATOM RATIO = 62000
241-PU ATOM RATIO = 3900
242-PU ATOM RATIO = 350
241-AM ATOM RATIO = 200
DAYS SINCE ISOTOPIC DETERMINATION 2
WATTS SAMPLE = 1.
THERMAL POWER ERROR = 0.150 %

} Input Data

* PLUTONIUM REPORT SAMPLE 1 WATT *

MASS RATIOS AT CAL DATE:
238-PU = 0.000107 +/- 15.00%
239-PU = 0.937506 +/- 1.00%
240-PU = 0.058369 +/- 15.00%
241-PU = 0.003686 +/- 3.00%
242-PU = 0.000332 +/- 30.00%
241-AM = 0.000190 +/- 7.00%

WATTS PER GRAM = 0.002318
GRAMS OF PLUTONIUM = 431.4 +/- 12.2

Presently, since the gamma-ray isotopic ratio measurements are not yet accurate enough to meet the goals of the Plutonium Inventory Verification Program (i.e., an overall uncertainty of less than 1% for calorimetric assay), radiocounting and mass spectrometry are used for the $^{238}\text{Pu}/^{239}\text{Pu}$ and the $^{240}\text{Pu}/^{239}\text{Pu}$ ratios.

In order to obtain a totally nondestructive assay with a 1% uncertainty it will be necessary to obtain all of the

isotopic ratios to within 3%. This goal has been met in the measurement of the $^{241}\text{Pu}/^{239}\text{Pu}$ ratio and is close to being met in the $^{241}\text{Am}/^{239}\text{Pu}$ ratio measurement.

Efforts to improve the measurement technique are continuing and are focused around the main areas of computer analysis, background reduction, and the use of different gamma-ray peak pairs.

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