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An Optical Emission Spectrographic Method for the Determination of Pu-240 in Plutonium Materials

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By
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THE DOW CHEMICAL COMPANY

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DENVER, COLORADO

U.S. ATOMIC ENERGY COMMISSION CONTRACT AT (29-1)-1106

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AN OPTICAL EMISSION SPECTROGRAPHIC METHOD
FOR THE DETERMINATION OF Pu-240 IN PLUTONIUM MATERIALS

by

E. Vejvoda

CLASSIFICATION CANCELLED

DATE 8-11-64

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ABSTRACT

An optical emission isotopic spectrographic method has been developed for the determination of Pu-240 in plutonium dioxide.

ACKNOWLEDGMENTS

The original suggestion for developing this method was made by J. T. Byrne.

The mass spectrometric analyses were performed by R. L. Carpenter.

The author wishes to acknowledge the laboratory assistance of A. J. Johnson and the assistance of D. W. Steinhaus in making the wavelength measurements.

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1. INTRODUCTION

The isotope shift for the plutonium spectrum has been reported by several investigators (Ref. 1, 2, 3, 4). Their findings suggested the possibility of developing a spectrographic method for the determination of Pu-240.

The primary problem was, of course, the resolution of the Pu-240 shift in the presence of high concentrations (>90%) of Pu-239. Preliminary resolution experiments with an Ebert spectrograph in the 6th order were successful in resolving the isotope shift for Pu-238, Pu-239, and Pu-240. As reported by Conway and Fred (1), the 4021 A line exhibited the best shift for the application of routine photometric procedures. From these preliminary findings, a spectrographic method was subsequently developed for the determination of Pu-240 in plutonium dioxide.

2. SUMMARY

An optical emission spectrographic method has been developed for the determination of Pu-240 in plutonium based materials. The analysis is based upon the resolution of the isotope spectral shift for Pu-240 using a plane grating spectrograph in the 6th order with a 15,000 LPI grating. The method is applicable to plutonium materials which can be converted to plutonium dioxide.

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The plutonium sample was converted to PuO_2 which was mixed with an equal weight of graphite. A small quantity of the graphite-plutonium dioxide mixture was excited with an a-c arc and the plutonium spectrum was recorded on a photographic plate. The isotopic resolution was accomplished using the Pu-4021A line, whereby the intensity of the Pu-240 component was related to Pu-240 content. The usual emulsion calibration working curve technique was followed to prepare the analytical curves using the line pairs of $\frac{\text{Pu-240 } 4021.48\text{\AA}}{\text{Pu-239 } 4021.63\text{\AA}}$ and $\frac{\text{Pu-240 } 4021.48\text{\AA}}{\text{Pu } 4026.72\text{\AA}}$. For the 4026.72A line, no detectable isotope shift was observed. Calibration for Pu-240 content was based on mass spectrometric values.

The standard deviation for 38 determinations on the same sample was $\pm 0.11\%$ Pu-240 with a coefficient of variation of 1.9 percent. The average difference (mass minus emission) for 76 samples was $+0.02\%$ Pu-240 with a standard deviation of $\pm 0.14\%$ Pu-240.

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3. EXPERIMENTAL

3.1 Sample Preparation

A sufficient amount of plutonium metal or plutonium salt (200-300 mg) was placed in a porcelain crucible and air oxidized to PuO_2 by heating in a furnace for one-half hour at 800 C. One-hundred mg of graphite and 100 mg of the PuO_2 sample were placed in a plastic vial and were thoroughly mixed by shaking the vial on a Wig-L-Bug (a dental amalgamator) for three minutes. The sample was loaded into the electrode crater by pushing the electrode down several times into the graphite-plutonium dioxide mixture until the crater was completely filled. The electrodes were loaded in quintuplet, thereby providing five spectra for each sample.

3.2 Standard Preparation

No standard preparation was required. Appropriate samples were selected for preparing the analytical curves based on their Pu-240 concentration as reported by mass spectrometry. The selected samples covered a concentration range of 2.30 to 6.50% Pu-240.

3.3 Electrode System

1. Lower Electrode - UCP Type 781, 1/8" diameter graphite rod with an under-cut crater of 1/16" diameter by 1/8" deep.
2. Upper Electrodes - UCP Type 1346B, pointed 1/8" diameter graphite rod, 1-1/2" long.

3.4 Apparatus

1. Spectrograph - Jarrell-Ash 3.4 meter Ebert mount plane grating instrument with a crossed cylindrical condensing lens system.
2. Microphotometer - Jarrell-Ash Recording Microphotometer with Bristol Recorder.
3. Source Unit - N.S.L. "Spec. Power" Unit
4. Photoprocessor - JACo Plate Processing Unit
5. Plotting Board - A.R.L. Calculating Board

3.5 Excitation

1. Source - a-c arc (not ignited)
2. Current - 4.5 amperes
3. Voltage - 2400 volts

3.6 Recording

1. Spectral Region - 4000-4060A 6th Order
2. Photographic Plate - Eastman SA-1 Emulsion

3.7 Spectrograph Parameters

1. Grating - 15,000 grooves/inch having a reciprocal linear dispersion of 5.0A/mm in the first order.
2. Angle of Incidence - 953.65° unblazed side, set for 24500A first order.
3. Slit Tube - +0.5 mm, rotated to the left for correct spectrum tilt.

3.8 Exposure Conditions

1. Slit Width - 10 microns
2. Slit Length - 1.6 millimeters
3. Filters - Glass
4. Analytical Gap - 2 millimeters
5. Pre-arc - 20 seconds
6. Exposure - 120 seconds

3.9 Plate Processing

1. Developer - 5 minutes with agitation
2. Short Stop - 20 seconds
3. Fixer - 5 minutes with agitation
4. Wash - 5 minutes with running water
5. Dry - Circulating warm air

3.10 Analytical Line Pairs

1. $\frac{\text{Pu } 4021.48\text{A}}{\text{Pu } 4021.63}$
2. $\frac{\text{Pu } 4021.48\text{A}}{\text{Pu } 4026.72\text{A}}$

3.11 Photometry

1. Microphotometer Slit - 5 microns by 1.2 millimeters
2. Emulsion Calibration - two-step preliminary curve method
3. Spectral Line Transmittances - recorded from microphotometer tracing and converted to relative intensity ratios using an A.R.L. Calculating Board.

3.12 Determination of Results

The Pu-240 results were obtained by employing the usual emulsion calibration-working curve technique and were reported in atom percent. No background corrections were applied.

4. RESULTS

4.1 Comparison of Results

Seventy-six samples were analyzed over a concentration range of 2-6.5% Pu-240 and the results compared with the values obtained by mass spectrometry. The average difference (mass minus emission values) for the 76 samples compared was +0.02% Pu-240. Since the standard deviation of the difference was $\pm 0.14\%$ Pu-240, the difference is not statistically significant at the 95% confidence level.

4.2 Precision

For reproducibility data, 38 spectra of the same sample were recorded on nine separate plates over a three-week period. This sample was used as a control standard. The standard deviation for the 38 determinations was $\pm 0.11\%$ Pu-240 with a coefficient of variation of 1.91%. The standard deviations and plate distribution for the 38 spectra are summarized in Table I. The tabulation of data for Table I is listed chronologically, consequently, the improvement in standard deviation for the last plate suggests a refinement in technique.

TABLE I
Reproducibility Data for a Single Sample

No. of Plates	No. of Det'rm.	% Pu-240 M-Spec	% Pu-240 E-Spec	S.D. (a)	C.V. (b)
1	9	5.84	5.71	$\pm 0.10\%$	1.75%
7	21	5.84	5.80	$\pm 0.12\%$	2.07%
	(3/plate)				
1	8	5.84	5.76	$\pm 0.07\%$	1.22%
-	-	----	----	-----	-----
9	38	5.84	5.77	$\pm 0.11\%$	1.91%

(a) Standard deviation defined as

$$S.D. = \sqrt{\frac{N\sum x^2 - (\sum x)^2}{N(N-1)}}$$

(b) Coefficient of variation defined as C.F. =

$$\frac{S.D.}{E.Spec.value} \times 100$$

5. DISCUSSION

5.1 Resolution

The chief problem of an isotopic emission method is the resolution of the isotopic shift to permit photometry of the individual isotope lines. In 1953, Conway and Fred⁽¹⁾ published their data on the isotope shift for Pu-238, Pu-239, Pu-240, and Pu-242. The wavelength separations assigned by them for the 4021A line are shown in Figure 1. To separate the Pu-239 and Pu-240 lines for microphotomentering, a spectrograph with a reciprocal linear dispersion of 0.6A/mm was required. The dispersion requirements were satisfied by employing an Ebert spectrograph in the 6th order. Microphotometer tracings (Figures 2 and 3) of the 4021A line photographed in the 6th order illustrate the resolution of Pu-238, Pu-239, Pu-240, and Pu-241 components.

Figure I

Isotope Shift for $\text{Pu}_{4021\text{A}}$

Figure 2

Isotope Resolution

Figure 3

Isotope Resolution

5.2 Sensitivity and Detection

Although the resolution and dispersion requirements were satisfied using the blazed side of the grating in the 6th order, the sensitivity and detection limits were poor. These deficiencies were overcome by rotating the grating and using the unblazed side of the plane grating. The lower detection limit for the method is about 2.0% of Pu-240. Attempts to gain sensitivity and lower the detection limit by increasing the exposure time and superimposing two exposures without racking the plate were unsuccessful. At lower concentrations the Pu-240 component suffered severe interference from the adjacent Pu-239 component while at higher concentrations the intensity ratios remained the same.

5.3 Reproducibility Data

Reproducibility evaluations were made for various line pairs. Two line pairs, $\frac{4021.48A}{4026.72A}$ and $\frac{4021.48}{4021.63}$, were selected based on their performance. The internal standard line 4021.63A was established as a Pu-239 component while the 4026.72A line demonstrated no detectable isotope shift. The intensity of the 4026.72A line was not affected by varying the concentration of Pu-239 as was the 4021.63A line. For the samples analyzed, the Pu-239 isotope varied over a concentration range of 93-98% and was generally inversely proportional to the Pu-240 content. The Pu-239 variance did not seem to effect the results as better sample precision and sample-to-sample reproducibility were obtained using the 4021.63A line for

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internal standardization. As a comparison, the standard deviation for the mass-emission differences on 76 samples was $\pm 0.20\%$ Pu-240 for the line pair of $\frac{4021.48A}{4026.72A}$ and $\pm 0.14\%$ Pu-240 for the line pair of $\frac{4021.48A}{4021.63A}$. For the single sample reported in Table I, the standard deviation was $\pm 0.32\%$ Pu-240 for the line pair of $\frac{4021.48A}{4026.72A}$ compared to $\pm 0.11\%$ Pu-240 for the line pair of $\frac{4021.48A}{4021.63A}$. The Pu-240 emission results used in the mass-emission comparison were determined using the $\frac{4021.48A}{4021.63A}$ line pair. The better reproducibility of $\frac{4021.48A}{4021.63A}$ may be due to less photometric error as both the internal standard line and analytical line are recorded consecutively with the same microphotometer sweep. The line pair of $\frac{4021.48A}{4026.72A}$ has been chiefly used for the determination of Pu-240 in samples with high concentrations of Pu-238 and Pu-240. In these samples the 4021.63A line is very weak and, in some instances, not detectable.

5.4 Hyperfine Structure (hfs)

In high order work, the hyperfine structure for a given spectral line can be troublesome in view of possible interference and profile patterns. The hyperfine structure for Pu-239 has been reported by several investigators (Ref. 5, 6, and 7). Under high dispersion, a large percentage of the more intense lines were observed by these investigators to be doublets with about the same intensity. Vanden Berg and Klinkenberg (5) reported a doublet structure for the 4021.A line,

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whereas Conway and Fred did not. However, the spectrograph used by Conway and Fred probably lacked the required dispersion to resolve the doublet structure. Likewise, no hfs was detected with our instrument for the 4021A line. Consequently, no interference was contemplated from the hfs of Pu-239.

5.5 Arc Intensity and Performance

The analytical line 4021A has been classified (5) as a Pu II line with an assigned relative arc intensity of 50. To excite the Pu II spectrum, both the a-c and d-c arcs were tried. The a-c arc was selected over the d-c arc based on spectral performance. The electrode geometry was not favorable for d-c excitation as the small electrode charge was consumed much too rapidly. The electrode size was considered critical and was kept to a minimum for better control over arc wandering. For the first 60 seconds of the arcing period, the electrode charge and the walls of the electrode crater are consumed at the same rate. After 60 seconds of burning, a small round ball of plutonium is formed. The arc seems to focus on the metal ball and wanders very little. The ball is gradually consumed by the arc. A moving plate of the arcing period is shown in Figure 4. The plutonium excitation is fairly constant throughout the 120-second exposure period.

Figure 4

5.6 Wavelength Assignments

The wavelengths of the plutonium lines used in this method were assigned from data compiled by McNally (8). As evident from McNally's report, there is a lack of agreement in the assigning of wavelengths for the plutonium spectrum. The wavelength of the Pu-240 line was taken as reported by Conway and Fred (1), while the wavelengths of the internal standard lines were taken as reported by Stukenbroeker and McNally (8). The assignment of these wavelengths was based on eyepiece measurements using iron for a reference.

For comparison purposes and to corroborate the eyepiece measurements, wavelength measurements were made using special equipment developed at Los Alamos by Steinhaus (9). The measurements were made from plutonium spectra photographed with the same Ebert spectrograph employed for the analysis. Uranium was used for a reference with wavelength values based on unpublished data by Steinhaus. The wavelength values are shown in Table II which also includes the published (8) wavelength values. The comparison in Table II indicates no gross errors which could effect the analytical application by erroneous spectral line identification.

TABLE II
Analytical Wavelength Values

<u>Line Description</u>	<u>Measured Wavelengths in Air</u>	<u>Published Wavelengths in Air</u>
Analytical Pu-240 Line	4021.55A	4021.48A
Internal Standard Line	4021.61A	4021.63A
Internal Standard Line	4026.68A	4026.72A

5.7 Advantages

The chief advantage of the emission procedure is that any plutonium based material can be analyzed which can be converted to plutonium dioxide. Besides metal samples, the method has been employed to analyze plutonium nitrate, plutonium peroxide, plutonium tetrafluoride, and plutonium dioxide materials. The method is somewhat faster than mass spectrometry as six samples per shift (8 hours) per man can be analyzed compared to three or four per shift per man for mass analysis using a CEC 21-220 instrument modified for solid analysis. With proper scheduling, two men could do 12 samples per shift with one emission spectrograph.

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