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# PALM CONTENT OF PRODUCTION METAL

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# PALM CONTENT OF PRODUCTION METAL

## Introduction

The purpose of this study was to determine the neptunium$^{237}$ input to the Purex process. Neptunium was isolated by solvent extraction, alpha counted for content, and checked for purity by alpha pulse height analysis. Standards were analyzed to determine sampling and chemical method efficiency.

## Summary

The neptunium$^{237}$ content of 639 g plutonium/ton uranium irradiated fuel was found to be 1.78 ± .09 g/ton of uranium at the 95 percent confidence level. Standard recovery for the chemical method was 96.7 percent and 98.0 percent for the sampling.

## Experimental

Chemical separation was made by extraction [1] in 0.5 M TTA in xylene. Several modifications to this method were necessary due to high radiation levels with a corresponding fission product contamination.

Sample size of the virgin metal solution was limited to 200 lambda by the radiation level from the sample carriers. To obtain a good counting rate on the final mount, it was necessary to combine the neptunium$^{237}$ content of two 200 lambda samples after a decontamination operation. A low beta activity on the mount is required for accurate alpha pulse height analysis, so no more than two 200 lambda aliquots could be combined.

Each 200 lambda sample was dispensed by the sampler into a vial containing hydroxylamine HCl and ferrous sulfamate. This quantitatively adjusted the neptunium valence to the IV state. A TTA extraction, constituting the primary decontamination step, was performed in the carrier and the organic phase was saved, using a phase separation bulb[2].

The neptunium was stripped from the organic phase into 8 M $HNO_3$. Following this step the two 200 lambda samples were combined. Zirconium-niobium was removed by contacting the 8 M $HNO_3$ with fresh TTA. The remainder of the separation follows the method outlined in the reference [1], consisting of a plutonium decontamination step and final isolation of the neptunium.

Each disc was counted on three alpha simpson proportional counters and an average count determined. In no case did the net count vary more than 3 percent between instruments. Each instrument had been checked and found to be within the 95 percent confidence level with a geometry standard. The beta content of the discs, in no case greater than 20 mr, was well below the beta threshold of the instruments. A twenty-minute alpha pulse height analysis was made of each disc and, where necessary, a correction was made. At the counting rate of these discs, it is not possible to designate over 95 percent neptunium content by alpha analysis. All discs with greater than 95 percent neptunium were considered to contain 100 percent.

Table I contains the data for the nine batches of dissolver solution analyzed. The average c/m column represents a 700 lambda mount of a one ml organic phase containing all the neptunium in the original 400 lambda of sample.

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## TABLE I

## ANALYTICAL SUMMARY - D5 METAL SOLUTION

| Date Batch | Average c/m (A.S.P.) | % $Np^{237}$ Alpha Analysis | mg $Np^{237}$/ gal | g $Np^{237}$/ Ton U | lb U/gal | Total alpha c/m/gal | g Pu/Ton U |
|---|---|---|---|---|---|---|---|
| 10-19-59 A | 248(a)(b) | 78.6 | 3.27 | 1.60 | 4.10 | ---- | ---- |
| 10-21-59 B | 178(a) | >95 | 3.44 | 1.76 | 3.90 | $8.36 \times 10^{10}$ | |
| | 174(a) | >95 | 3.36 | 1.72 | 3.90 | $8.36 \times 10^{10}$ | 546 |
| 10-22-59 C | 175 | >95 | 3.00 | 1.53 | 3.93 | $1.01 \times 10^{11}$ | 653 |
| 10-23-59 D | 217 | >95 | 3.73 | 1.78 | 4.19 | $1.03 \times 10^{11}$ | 622 |
| 10-26-59 E | 183(c) | 94 | 3.32 | 1.66 | 4.00 | $1.02 \times 10^{11}$ | |
| | 177(c) | >95 | 3.42 | 1.71 | 4.00 | $1.02 \times 10^{11}$ | 647 |
| 10-29-59 F | 261.5 | 85.5 | 3.84 | 1.87 | 4.11 | $1.09 \times 10^{11}$ | |
| | 108(d) | >95 | 3.71 | 1.81 | 4.11 | $1.09 \times 10^{11}$ | 656 |
| 11-2-59 G | 247 | 88 | 3.73 | 1.66 | 4.50 | $1.09 \times 10^{11}$ | |
| | 239 | 89 | 3.65 | 1.62 | 4.50 | $1.09 \times 10^{11}$ | |
| | 247 | 91 | 3.86 | 1.68 | 4.59 | $1.12 \times 10^{11}$ | |
| | 252 | 90 | 3.89 | 1.69 | 4.59 | $1.12 \times 10^{11}$ | |
| | 227 | 94 | 3.66 | 1.63 | 4.50 | $1.07 \times 10^{11}$ | |
| | 208 | >95 | 3.57 | 1.59 | 4.50 | $1.07 \times 10^{11}$ | 630 |

Notes:

(a) Due to a later method modification, which was measurable, 11 percent of the available neptunium was lost. This correction was made in the mg Np/gal column.

(b) 800 lambda of the neptunium phase was mounted.

(c) Bad reagent was detected by a standard run simultaneous with the sample. A correction of $\frac{.967}{.86} = \frac{1}{.889}$ was incorporated in the mg Np/gal column.

(d) A 700 lambda mount of a 200 lambda dissolver sample.

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Standard recoveries for the chemical analysis, using a neptunium$^{237}$ standard prepared in a synthetic dissolver matrix, were as follows:

| Date | Percent Recovery |
|---|---|
| 10-22-59 | 98.7 |
| 10-23-59 | 94.1 |
| 10-28-59 | 96.9 |
| 11-5-59 | 97.2 |

The average recovery was 96.7 percent. Standard recoveries for the sampler were measured by dispensing 200 lambda aliquots of 8 N $HNO_3$ into 5 ml of distilled water. The resulting $HNO_3$ concentration was determined in the laboratory, using a caustic titration.

| Date | Percent Recovery |
|---|---|
| 10-29-59 | 97.3 |
| 11-4-59 | 98.6 |

The average recovery was 98.0 percent.

Discussion

The data from Table I was evaluated by Operations Research and Synthesis Operation, Hanford Laboratories Operation. According to this statistical evaluation, the neptunium concentration of the 639 g plutonium/ton uranium production fuel is 1.69 ± 0.09 g/ton of uranium at the 95 percent confidence level with no bias correction. Also, according to this evaluation, each single batch value is as good as the multiple value.

The bias created by chemical analysis and sampling is calculated to be equal to:

$$(.98)(.967) = .948$$

At the 95 percent confidence level, the irradiated fuel elements contain:

$$\frac{1.69 \pm .09}{.948} = 1.78 \pm .09 \text{ g/ton of uranium}$$

The IPD predicted g plutonium/ton averaged 632 for the metal charged to the dissolvers during this study as compared to 639 average determined from D5 samples.

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