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MEASUREMENTS OF REDOX PRODUCED NEPTUNIUM

by

J. S. Buckingham

Separations Chemistry Laboratory
Research and Engineering Operation
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MEASUREMENTS OF REDOX PRODUCED NEPTUNIUM

INTRODUCTION

All of the neptunium recovered in the Redox and Purex Separations plants has been purified in a pilot plant located in the 222-S laboratory building. In the Redox and Purex plants the unpurified or crude neptunium was sampled for operational control, loaded into PR cans and transported to the pilot plant. At the pilot plant, the contents of the PR cans were transferred into two 100-liter feed tanks equipped with float gauges calibrated in 1-liter increments. Because of occasional large discrepancies between Redox and Purex load-outs and the pilot plant measured receipts, the pilot plant receipts were taken as the official SS material measurements. With the completion of the neptunium purification facility in the Purex plant (Q cell), the pilot plant facility will no longer be used. Redox crude neptunium will be sent to Purex for purification. It will not be possible to composite and sample the Redox plant load-outs in the Purex plant Q cell with the present equipment as was done in the pilot plant. Therefore, either accurate load-out measurements will be required at the Redox plant, or accurate receiving measurements will be needed at Purex.

In February, four batches of neptunium were recovered from the Redox plant and sent to the pilot plant for further purification. Load-outs from the Redox plant and receipts in the pilot plant were both measured and are shown in the body of this report.

SUMMARY AND CONCLUSIONS

1. The reliability of neptunium measurements in the E-4 tank is not good enough for SS material control. Future studies of E-4 tank measurements may indicate the process controls necessary to improve the reliability of the measurements to the point where they may be used for SS material control.
2. Initial neptunium load-outs from the Redox plant should be determined by sampling each can.
3. A 20 to 30 gallon tank capable of holding a complete batch of Redox neptunium should be installed either between the Redox L-2 concentrator and the L-21 load-out tank or in the Purex plant Q cell. This tank could then be used to measure each complete batch of unpurified neptunium recovered in the Redox plant.
4. The concentration of the neptunium in samples should be determined by spectrophotometric means and verified by alpha counting techniques. A standard simulating Redox neptunium load-outs should be run on a routine basis to establish a bias correction for the analytical methods.

DISCUSSION

Four batches of neptunium were recovered in the Redox plant and sent to the ion exchange purification pilot plant during February 1963. The batch number, the can numbers, the date, the point in Redox where the material was measured, and the amount of neptunium and plutonium sent are shown in Table I below. The volume and amount of material received in the pilot plant feed tanks are also shown.

[redacted]

TABLE I

NEPTUNIUM SENT FROM REDOX TO NEPTUNIUM PURIFICATION PILOT PLANT

Date	Run	Cans	Measured	Sent		Received		
				Np	Pu	Vol.	Np	Pu
2-4-63	1	1 thru 8	E-4	785.1	10.7	52	785	3
2-5-63	2	9 thru 14	E-4	680.9	13.2	48	706	6
2-11-63	3	15 thru 25	E-4	479	5	86	533	6
2-26-63	4	26 thru 36	cans	651	164		651	163
Total				2596	192.9		2775	178

The amount of neptunium measured in E-4 and received in the pilot plant checked very closely on the first batch; however, with the seond batch approximately 4 per cent more was received than was sent, and with the third batch, 11 per cent more was received. Measurements of neptunium in E-4 would be adequate for process control, but would not be reliable enough for SS material control. Further studies of E-4 tank measurements may show ways to improve the reliability of the E-4 measurement and subsequent concentration and load-out to the point where they may be used for SS material control. A comparison of the amount of neptunium as determined by a thief sample from each PR can and the amount of neptunium received in the pilot plant showed excellent agreement.

The disadvantages of determining the amount of neptunium produced by analyzing thief samples from each PR can are: 1) each batch would require from 6 to 12 samples. 2) There is no assurance that

the solution in the PR can is homogeneous. 3) Analytical errors may be additive. The installation of a 20 to 30 gallon tank equipped with a volume measuring device either between the Redox concentrator L-2 and the load-out tank L-21 or in the Purex Q-cell would allow an entire batch of Redox material to be composited before being sampled, eliminating the necessity for so many thief samples. By equipping the tank with a sparger or an agitator, homogeneity of the solution could be assured.

A bias in analytical methods could be detected by analyzing a quality control standard similar to the Redox material along with the process sample. The regular quality control standard and referee program could be adapted to cover Redox neptunium product.