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LABORATORY EVALUATION OF NELCO CALCIUM FOR

PLUTONIUM TETRAFLUORIDE REDUCTION

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R. L. Beede

~~By Yellow Files~~

234-5 Development Unit
Separations Technology Section
ENGINEERING DEPARTMENT

September 15, 1955

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INTRODUCTION

This laboratory evaluation was conducted to see if the calcium as supplied by New England Lime Company (Nelco) would produce plutonium metal of satisfactory purity and in good yield, since the use of the calcium as supplied by Nelco

- would save ca. \$6.00 per pound.

SUMMARY

Samples of a 50 pound shipment of New England Lime Company, Nelco, -4 +80 mesh calcium, were tested in two 1200 g scale reductions. Plutonium metal of normal purity resulted, and reduction yields were 95.4 and 98.0 per cent.

EXPERIMENTAL

First, small (60 g scale) reductions were made in the laboratory, both with re-distilled (ORNL) -8 +80 mesh and with Nelco -4 +80 and -8 +80 mesh calcium. Plutonium metal of normal purity and satisfactory yields were obtained. Then, two 1200 g scale reductions were made with the Nelco -4 +80 mesh calcium, without iodine booster, and reduction yields of 95.4 and 98.0 per cent were obtained. Mixing of the charge was done by hand, the charge was packed by hand, and the pressure vessel was sealed manually. The vessel was pressurized to 90 psi, leak tested with argon, and the argon discharged into the hood atmosphere before firing. Firing time for the two large runs with the 10 KW induction unit was two minutes longer than normal, or 26 minutes per run. The data on these runs are recorded in Table I.

DISCUSSION

The calcium received from ORNL in the past has been ground and graded to -8 +80 mesh calcium in the 234-5 Building at a minimum cost of ca. \$0.90 per pound. The calcium as supplied by Nelco is -4 +80 mesh. This material was tested as received since elimination of grinding and grading could represent an additional savings. The use of the coarser -4 +80 mesh calcium produces more segregation of plutonium fluoride and calcium mixture than with -8 +80 mesh. This segregation has little effect on reduction yields as was demonstrated by two laboratory 1200 g scale reductions⁽¹⁾, DLY-15-6-1 and DLY-15-6-2, in which the plutonium fluoride and -8 +80 mesh calcium, no iodine booster, were charged in alternate layers, and plutonium metal yields of 94.0 and 99.1 per cent were obtained. No change in button characteristics or button to slag or button to crucible sticking was evident.

The magnesium content, 1000 ppm, of this shipment of Nelco calcium is lower than that indicated by the vendor, namely, 5000 ppm. The presence of this higher amount of magnesium is not expected to affect the final plutonium button purity.

(1) Notebook HWN-930.

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TABLE I

Results of 1200 g Scale Reductions of PuF₄ with
Nelco -4 +80 Mesh Calcium

	<u>DLY-15-7-1</u>	<u>DLY-15-8-1</u>
Per Cent Yield	95.4 ⁽¹⁾	98.0
<u>Element</u>	<u>Analyses</u>	<u>Analyses</u>
F	10.9	7.8
C	200	150
Al	30	< 20
B	20,5 ⁽²⁾	< 1.0
Be	< 0.02	< 0.02
Ca	16	< 3
Cr	50	50
Fe	200	1000
K	20	
La	< 30	< 30
Ni	< 1	< 1
Mg	100	100
Mn	100	200
Na	100	2
Ni	100	50
Si	20	50

(1) This was an off-color fluoride of 19.5 per cent fluorine.

(2) High boron a result of talc contamination.

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TABLE II

Spectrochemical Analyses of Calcium

<u>Element</u>	<u>Specification for Calcium</u>	<u>Nelco Calcium</u>	<u>Redistilled⁽²⁾ Calcium</u>
Al	10 ⁽¹⁾	< 10	< 10
B	< 1	< 1	< 1
Be	< 1	< 1	< 1
Co	10	< 10	< 10
Cr	10	< 10	< 10
Cu	10	10	10
Fe	10	10	100
Li	1	< 1	< 1
Mg	1000	1000	100
Mn	10	100	10
Na	10	< 10	< 10
Ni	10	< 10	< 10
Si	10	10	10

(1) Parts per million parts calcium.

(2) From distillation facility at ORNL.

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