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Precipitation Studies
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SUBJECT Metathesis of Carrier-Free

Potassium Plutonium Fluoride

To L. Perlman

FROM L. B. Warner

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1. Nathan
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I. Perlman

L. B. Warner

Metathesis of Carrier-Free Potassium Plutonium Fluoride

In lieu of the H_2SO_4 fuming procedure for getting potassium plutonium fluoride into solution, KOH metathesis with HNO_3 dissolving has been used on Isolation Batch 107. Metathesis has several inherent advantages over fuming: (1) Elimination of fuming as a health hazard due to possible body contact, especially breathing of spray; (2) Less loss of material due to spattering during fuming; (3) Possibility of keeping the Pu free of sulfate ion. Sulfate has proved very troublesome in the later steps of isolation. It is very difficult to free the Pu of sulfate, and if present is likely to cause precipitation of Pu sulfate when acid concentrations are reduced and dilutions made; (4) Fuming samples of fluoride greater than 1 or 2 grams tends to become a time consuming operation, and possibly an impractical method for handling large samples. For these reasons, it was decided to use a KOH metathesis. Isolation Batch 107 was received from V. R. Cooper's group as a potassium Pu fluoride precipitate containing ~1.5 gms Pu. No LaF_3 had been precipitated with the product. The fluoride precipitate was treated 2 times with 20 ml of 10 M KOH at $95^\circ C$ for 1/2 to 3/4 hour each treatment. Motor agitation was used. The sample was then given 4- H_2O washes of ~40 ml each. After this treatment the precipitate appeared to be dense and light brown in color; it dissolved slowly in concentrated HNO_3 to give an amber colored solution. After centrifuging, a small amount of black residue remained undissolved and some black oily precipitate floated on top. The combined amount of product associated with these insoluble residues was 0.06% of the total. The total amount of product lost in the combined metathesis liquid plus washes was 0.1% of the total. These operations were carried out in glass. During subsequent isolation steps a considerable amount of silicic acid separated out. This would suggest the advisability of using platinum or stainless steel equipment for the metathesis.

LBW/dos

L. B. Warner

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For The Atomic Energy Commission
H.F. Canale
Chief, Declassification Branch ml

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