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RALA PROCESS RESEARCH

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I. STATUS OF PROJECT

This project was authorized for the development of new chemical processes for the production of RaLa sources for use in the new Ten-site Laboratory. In the early phases of this work many processes were investigated and several found to be promising. When time deadlines required that engineering design work on particular processes be started, the two showing the most promise were selected and all effort was concentrated on them. This does not mean that other methods cannot be used, but the remote control process cells will be equipped initially for the operation of either of these two processes. Other methods will require modification of the equipment in minor details or addition of other equipment.

These two processes, the hydroxide process and the nitric acid process, differ in the reagent used for the initial separation of lanthanum from barium. Both depend upon centrifugation for phase separation and both utilize a second step in which the final lanthanum precipitate is obtained as a fluoride. Both processes have been tested thoroughly on a tracer scale. A separate project has been authorized for testing at a high level of activity. Present work consists of study of a number of details brought out by the engineering study of theprocesses, such as the most favorable volume, reduction of mechanical loss of precipitate, and the effect of temperature of the solutions.

II. SUMMARY OF PRESENT WORK

Determinations of per cent barium impurity found in the production of lanthanum sources have been continued.

Temperature measurements of highly active solutions have been made and plotted in an effort to predict the temperature anticipated when solutions containing 10,000 curies are processed.

Additional studies have been carried out on the proposed processes and further runs at the DP mock-up.

Quantities of sodium which can be tolerated in future processes have been calculated.

It has been shown in tracer and high level runs that it is necessary to dissolve completely the barium nitrate precipitates before obtaining complete lanthanum recoveries.

A method has been worked out for wet-ashing filter paper used in collecting plutonium fluoride dusts.

A. Per Cent Barium Impurity Studies

Results obtained on shipments 42 and 43 are given below:

	Milking	Per Cent Ba, 1st Step	Per Cent Ba, Final Source
Shipment 42:	1st	10.1	2.50
	2nd	10.8	about 0.30
	3rd	11.4	0.19
	4th	- -	less than 0.0005$^{\Delta}$
Shipment 43:	1st	15.9	0.26
	2nd	1.3	- -
	3rd	2.8	- -

$^{\Delta}$Immediately after the third milking four hydroxide precipitations were made to remove cerium and other impurities. The source from the fourth milking was used in air absorption measurements and in the absolute counting experiment.

B. Temperature Measurements

The following data have been obtained on several different active solutions at Bayo; when these data were plotted on log-log paper and extrapolated to 10,000 curies, an approximate temperature of 90° C. above room temperature were obtained. This work is by no means final and will be continued.

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Volume of Solution	Curies Present	Degrees Cent. above Room Temp.
about 60 cc.	1370	19Δ
" 60 cc.	1350	20Δ
" 60 cc.	1065	20
" 60 cc.	1325	21Δ
" 40 cc.	1825	29
" 65 cc.	2090	34
" 45 cc.	3130	45
" 40 cc.	4400	53.4

ΔNot at maximum lanthanum growth.

C. Tracer Runs at DP Mock-up

Several tracer scale runs were made on the fuming nitric acid and hydroxide processes at the DP mock-up to check the chemistry and familiarize the operators in the new remote control techniques.

Favorable results were obtained with the hydroxide process. However, the low yields with the fuming nitric acid method instigated further development work.

It has been shown experimentally that to obtain a 95% yield on 40 mg. of lanthanum, the solution from which the lanthanum fluoride is precipitated must not exceed 150 cc. The decrease in yield obtained when this volume is exceeded is attributed to mechanical losses. Now if this volume is a limiting factor, the maximum barium (as barium *metal*) to be tolerated is 1.68 grams if dilution is used in adjusting the normality to optimum conditions. If, instead, one neutralizes a portion of the nitric acid with 3N base, then 2.14 grams could be processed.

D. Effect of Sodium on Proposed Processes

In answer to a request from Oak Ridge concerning the amount of sodium which can be tolerated in our proposed processes, the following results have been obtained:

1. Fuming Nitric Acid Process

No experimental evidence has been found for the formation of an insoluble double salt between lanthanum nitrate and sodium nitrate under operating conditions. However, because of solubility considerations, it is imperative that no more than 2 grams of sodium be present and the total barium not exceed 1.5 grams.

2. Hydroxide Process

As high as 20 grams of sodium (74 grams of sodium nitrate) can be tolerated without any deleterious effects.

E. Recovery of Lanthanum from Barium Nitrate Precipitate

The lanthanum which grows in while the barium is in the form of a precipitate can be completely recovered only when all of the barium nitrate has been dissolved. The results below show the per cent lanthanum which was leached from barium nitrate, in various nitric acid concentrations, during the growth of the daughter.

Nitric Acid	% La Recovered	Remarks
0.1	94	Precipitate completely dissolved.
0.5	98	" " "
3.0	79	Slight precipitate.
6.0	45	About 50% precipitate remained .
16.0	26	" 100% " "

When the 200 curie run in the test cell was made, 22% of the lanthanum remained within the barium nitrate crystals. About 1.9 grams barium nitrate in 12 cc. of 0.3N nitric acid was the starting solution.

F. Wet Ashing of Filter Paper

The Hollingsworth-Vose Filter Paper No. H-70 can be dissolved by treatment with concentrated sulfuric and nitric acids. Hydrofluoric acid is then used to dissolve the siliceous residue fillers, which constitute 13% by weight of the paper. The hydrofluoric acid is removed by evaporation with excess sulfuric acid. This method was developed for use in the air decontamination studies.