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PRECIPITATION OF CERIUM SULFATE

by

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Separations Chemistry Laboratory
Research and Engineering Operation
Chemical Processing Department

January 27, 1964

HANFORD ATOMIC PRODUCTS OPERATION
RICHLAND, WASHINGTON

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PRECIPITATION OF CERIUM SULFATEINTRODUCTION

A batch of cerium is being purified during January in the semiworks using D2EHPA as solvent. The cerium will be stripped from the solvent with nitric acid, precipitated as the sulfate, and slurried into the HAP0 1B-1 cask for shipment to Oak Ridge. H. H. Van Tuyl determined the optimum conditions for the sulfate precipitation of cerium purified by peroxy acetate precipitations; (1) However, because the composition of the cerium purified by D2EHPA is somewhat different, additional studies of cerium sulfate precipitation were made in the Separations Chemistry Laboratory.

SUMMARY

Cerium sulfate purified by D2EHPA in Semiworks can be precipitated by adjusting the pH of the solution to between 1 and 2 in tank 6 with 50% caustic. The solution can then be transferred through tank 1 to tank 67, where sodium bisulfate is added to make the solution 0.5M sulfate. A stoichiometric amount (mole for mole) of 50 per cent caustic is added to just neutralize the sodium bisulfate. The precipitate is digested one hour at 60 C, then filtered.

The pH of the final solution is critical for good cerium recoveries. Below a pH of 1, losses to the supernatant liquid increase. Above a pH of 7 a slimy precipitate forms which is difficult to filter. Sodium bisulfate acts as a monobasic acid exhibiting an extremely sharp pH break at the neutral point in a titration curve. Sodium bisulfate shows no buffering action; therefore, when adjusting to a pH of between 1 and 2 the caustic should be added slowly to prevent over neutralization. At pH's higher than 2, a precipitate forms, presumably cerium hydroxide which redissolves at lower pH's if no sulfate is present.

DISCUSSION AND RESULTS

Titration curves for 6M sodium bisulfate (Figure 1.) and a mixture of synthetic cerium product and sodium bisulfate (Figure 2) were run to determine the buffering capacity of sodium bisulfate. The titration curves show that sodium bisulfate or mixtures of cerium nitrate and sodium bisulfate have little or no buffering capacity, and the pH break is very sharp.

The precipitation of cerium as the sulfate was studied by precipitating portions of a synthetic cerium product tagged with Ce^{144} under varying conditions. The synthetic cerium product used was 3 g/l cerium and 2M HNO_3 . The results are shown in Table I.

TABLE IPRECIPITATION OF CERIUM AS THE SULFATE

<u>Temp., C</u>	<u>pH</u>	<u>SO₄⁼, M</u>	<u>Ce loss, %</u>
25	0	0.54	28.1
25	0.5	0.54	6.2
25	1	0.54	6.1
25	2	0.54	7.5
25	9.8	0.54	0.3
60	0	0.54	13.0
60	0.5	0.54	1.5
60	1	0.54	1.2
60	2	0.54	0.6
60	7	0.54	0.4
25	0	1	10.5
25	0.5	1	3.2
25	1	1	5.2
25	2	1	5.8
25	10.5	1	11.9
60	0	1	24.2
60	0.5	1	5.7
60	1	1	0.7
60	2	1	0.5
60	11.1	1	1.4

A number of experiments were run to determine the optimum conditions for the formation of an easily filtered cerium sulfate precipitate. In these experiments, it was observed that above a pH of 2 to 3 a precipitate formed in the cerium product, presumably cerium hydroxide. Not all of this precipitate redissolved when sodium bisulfate was added to the desired sulfate concentration even though the pH of the solution was reduced to less than 1.

Cerium sulfate precipitated at a pH of 7 or greater was "slimy" and tended to plug the filter. For example, the cerium sulfate formed at a pH of 1.9 filtered in 1.5 minutes, while the precipitate formed at a pH of 9 required more than 30 minutes to filter.

- (1) H. H. Van Tuyl, "Laboratory Studies of the Precipitation of Cerium Sulfate for Off-Site Shipment." Nov. 1, 1961, HW-71589.

Fig. 1

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