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UNITED STATES ATOMIC ENERGY COMMISSION

Progress Report For September 1949

RECOVERY OF THORIUM AND URANIUM FROM MONAZITE SANDS

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Contract No. AT 30-1-Gen-228

RECOVERY OF THORIUM AND URANIUM FROM MONAZITE SANDS

(A. E. Bearse and J. W. Clegg, Supervisors)

SUMMARY

A quarterly report (BMI-JDS-209) on the recovery of thorium and uranium from monazite sands has recently been completed, covering the work on this project up to August 15, 1949. The present report summarizes the work of the past month.

The study of the leaching of hydroxide precipitates with a solution of sodium carbonate and bicarbonate has been continued with the main objective of discovering the conditions necessary for high thorium recoveries.

The effect of concentration of several interfering anions on the adsorption of uranium by anion-exchange resins from carbonate solutions has been determined. Chloride ion was found to interfere more seriously than bicarbonate or hydroxyl ion.

Two runs with the laboratory production unit using hydrochloric acid to dissolve the hydrous oxide cake have been completed. Substantial progress has been made toward increasing the recoveries of the thorium and uranium. Losses of uranium to the caustic soda were reduced considerably in two runs.

Additional manpower is being applied to the monazite project in order to speed up the solution of many of the problems involved.

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SEPARATION OF THORIUM AND URANIUM FROM RARE EARTHS BY CARBONATE LEACHING

The investigation of the carbonate leaching of hydroxide precipitates to separate thorium and uranium from rare earths, iron, and titanium and to form a solution satisfactory for the anion-exchange separation of the uranium from the thorium has been continued.

A concentrated study of the leaching conditions necessary to obtain nearly complete recovery of the thorium and uranium is being made. The investigation is being carried out with a leach solution containing sodium carbonate and bicarbonate (see report BMI-JDS-208, August, 1949). Analytical data for the experiments which have been completed are not available at this time. It appears that the major difficulties in obtaining complete dissolution of the thorium from a hydroxide precipitate in the carbonate solution are:

- (1) The repulping and mixing of the hydroxide precipitate with the carbonate solution to obtain satisfactory contact between all of the thorium and a sufficient excess of carbonate;
- (2) The existence in the precipitate of forms of thorium more insoluble than the hydroxide, e.g., oxide or phosphate.

SEPARATION OF URANIUM FROM THORIUM BY ANION EXCHANGE

The study of the application of anion exchange to separate uranium from thorium in carbonate solutions has been continued.

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It was found in previous work (see report BMI-JDS-208, August, 1949) that certain anions which may be present in the carbonate solution interfere with the adsorption of uranium. An investigation of the effect of these anions on the capacity of Amberlite resin, IRA-400, for uranium in ten per cent sodium carbonate solutions is being made. The data for chloride, hydroxide, and bicarbonate anions are complete (see Table 28). These experiments were conducted by passing (at a rate of 200 milliliters per hour) the sodium carbonate solution containing 20-25 milligrams of uranium per 100 milliliters and the the interfering anion through a bed (1 by 30 centimeters) of Amberlite IRA-400. The results are presented in terms of the amount of uranium quantitatively removed from solution per 100 grams of resin (dry basis).

The data show that all of these anions (Cl^- , OH^- , and HCO_3^-) interfere with the adsorption of uranium. Chloride ions gave the most serious interference.

SOLUBILITY OF SODIUM PHOSPHATE IN SODIUM HYDROXIDE SOLUTIONS

The determination of the solubility of trisodium phosphate in solutions of sodium hydroxide at various temperatures is being continued in order to provide useful data for the operation of the monazite-caustic soda digestion process.

PLANS FOR FUTURE WORK ON CHEMICAL STUDIES

At present, hydrochloric acid is being used to dissolve the reaction mass obtained from the caustic soda treatment of monazite sand.

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TABLE 28. EFFECT OF EXTRANEEOUS ANIONS ON THE CAPACITY OF AMBERLITE IRA-400 FOR URANIUM IN TEN PER CENT SODIUM CARBONATE SOLUTIONS

Concentration of Extraneous Salt (g./100 ml. of Soln.)	Uranium Adsorbed Quantitatively (g./100 g. of Dry Amberlite IRA-400)		
	NaHCO ₃	NH ₄ OH	NaCl
0	5.53	5.53	5.53
0.1	4.26	4.48	4.00
0.5	3.26	3.48	0.42
1.0	2.58	3.27	0
3.0	0.58		
5.0	0.37		

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Since there is a serious corrosion problem with hydrochloric acid, it might be desirable to use sulfuric acid. An investigation of this possibility has been started.

In all of the experiments conducted to test the selective hydroxide separation at pH 5.5 of the thorium and uranium from the bulk of the rare earths, ammonium hydroxide has been used as the precipitant. A study of the possible use of sodium hydroxide for this purpose is being investigated.

The work on the carbonate leaching of hydroxide precipitates to recover thorium and uranium and to form a solution satisfactory for anion-exchange techniques will be continued.

The work on anion-exchange methods of separating uranium from thorium in carbonate solutions will be expanded. The effect of possible interfering anions will be determined. Other strong-base anion-exchange resins, such as Dowex 1, Dowex 2, Ionac A-301, Permutit S, and Cochranex AD will be evaluated.

LABORATORY PRODUCTION UNIT

During the past month, using the laboratory production unit equipment, two runs were made for which analytical data are available. For both of these runs, the time and temperature of the digestion periods prior to filtration of the hydrous oxide slurries were increased. After dilution of the reaction mixture formed when monazite sand was treated with a 60 per cent solution of caustic soda in the usual manner,

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the slurries were boiled (previously the procedure was to digest at about 170-200°F.) at a temperature of 240°F. for a period of time in excess of two hours. The slurries were then allowed to stand and cool without agitation until the temperature dropped to about 160°F., when filtration was begun. After filtration of the slurries and washing of the hydrous oxide cake was completed, the cake was removed from the filter and treated with 37 per cent hydrochloric acid in a rubber-lined tank. Twelve pounds of acid was used to treat the cake from 5 pounds of sand. During the treatment the temperature was held at about 200°F. After the cake had dissolved, the mixture was diluted somewhat and filtered through paper, using a large laboratory vacuum filter.

The analytical data obtained from the two runs indicated definite improvement in the uranium losses to the caustic filtrate, the loss having been only 17 per cent of the total in one case as compared to previous losses of from 30 to 50 per cent. The iron content of the filtrate was also reduced greatly. It has not been determined whether the favorable results were caused by the change in digestion treatment, or by the fact that the stainless steel reaction vessel was no longer being used for the nitric acid dissolution. The simultaneous low iron and low uranium content of the caustic filtrate appear to be closely related phenomena.

By modifying somewhat the evaporation and crystallization techniques used when separating the trisodium phosphate from the caustic filtrate, it was possible to hold most of the uranium in the caustic soda liquor to be recycled to the process, and thereby reduce the loss of

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uranium with the trisodium phosphate crystals to less than two per cent of the total present. This loss will be further reduced if the trisodium phosphate is recrystallized, as will probably be necessary in order to prevent excessively high losses of caustic soda with the phosphate crystals.

The dissolution of the hydrous oxide cake in hydrochloric acid led to considerably higher recoveries of thorium in the final acid solutions than had been found while using the nitric acid-hydrogen peroxide system. In one run, a thorium recovery of 88 per cent was obtained, which is almost twice any previous recovery figure observed for the nitric acid treatment.

While it appears that some headway is being made toward finding the solutions to the difficulties associated with the process, it is still too early to view any of the favorable results as conclusive. Not until enough data have been accumulated and the effects discussed above are fully reproducible under controlled conditions can the situation be considered well in hand.

Work will be continued along the general lines discussed above in order to gather the necessary information and experience, so that some definite plans can be made for scaling up the process to the point where a careful engineering study will be feasible.

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