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D. E. Ferguson

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DATE	MAR 12 1957 <i>jet</i>
For The Atomic Energy Commission	
<i>H. R. Canale</i>	
Chief, Declassification Branch	

SUMMARY

Mass-233 Studies

Proposed Thorex second cycle flowsheets gave additional decontamination factors of 10 or less for the pilot plant first-cycle product from the short-cooled material run. Previously successful methods of solvent treatment did not decontaminate the used pilot plant solvent from this run. Both iodate and hydroxide precipitations recovered over 99% of the protactinium from short-cooled AP.

Volatility Studies

The temperature dependence of the $UF_6 \cdot 3NaF$ decomposition rate to the U^{+5} form is expressed by the equation

$$\log r = 6.09 - 5.22 \times 10^3/T$$

where r is the decomposition rate in reciprocal minutes and T the absolute temperature. This expression was derived from data secured in fourteen runs over the temperature range of 245-355°C.

Thorex Second Uranium Cycle

The second cycle processing of Thorex uranium product was completed with the decontamination of 3000 g of U^{233} . A total of 20 second cycle runs were made during the last three months in this program. The uranium product met both fission product and ionic specifications in all except the two runs reported previously.

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Equipment Decontamination

Chromous sulfate solutions were satisfactorily prepared by electrolytic reduction of solutions containing 0.4 mole of either $\text{Cr}_2(\text{SO}_4)_3 \cdot \text{K}_2\text{SO}_4 \cdot 24\text{H}_2\text{O}$ or CrO_3 per liter of 1 M H_2SO_4 . Anodic and cathodic solutions must be separated by a diaphragm in order to prevent reoxidization of the chromous ion.

Homogeneous Reactor Blanket Processing

There was no PuO_2 precipitate formed by heating 1.4 g UO_2SO_4 at 250°C after any of four daily additions of plutonium which gradually raised the plutonium concentration to 21.3 mg/kg H_2O .

Zirconium oxide did not act as an effective carrier for PuO_2 from 1.4 g UO_2SO_4 at 250°C .

Simulated HRT core solids were more readily dissolved in H_3PO_4 after baking at 350°C for 15 min. Attempts to prevent zirconium hydrolysis in 4 M H_3PO_4 at 100°C were unsuccessful.

Scouting tests indicate that tantalum is suitable for containing phosphoric acid at 250°C .

Homogeneous Reactor Fuel and Blanket Studies

A 750 g Th/kg H_2O slurry of the standard oxide has been stirred at 300°C in the LITR for ten days. Testing of the bomb dismantling equipment in the "hot cell" has been initiated. The settling rate and settled bed density of the standard slurry were decreased by blending in the Waring blender at room temperature. Decreasing the addition rate of the thorium nitrate to an oxalic acid solution accelerated the settling rate of the resulting oxalate precipitate.

Homogeneous Reactor Chemical Processing

Digestion of simulated HRT chemical plant solids with NaOH and Na_2CO_3 solutions at 250°C did not render them soluble in dilute mineral acids. Mixtures of HNO_3 and HCl were not effective for dissolving such solids or leaching uranium from them.

The effect of gamma radiation on iodine valence state in UO_2SO_4 solution at 100°C is opposite to the effect at 250°C ; i.e., radiation at 100°C decreases the free iodine/oxidized iodine ratio, at 250°C it increases the ratio.

Dissolution Studies

The presence of HNO_3 in HF solutions slightly enhanced the dissolution rate of zirconium and titanium and apparently does not promote passivation.

Zircex

Zircex hydrochlorination can be used to remove Zircaloy-2 jackets from UO_2 cores without excessive chloride pickup by the UO_2 .

Mice

Hydrous ferric oxide scavenging appears to be technologically and economically feasible for removing non-ionized U from sols produced by vaporization of uranium metal under water. Precipitates were produced in solutions containing 50 mg Fe/liter by making them $\sim 3 \times 10^{-3} \text{ M}$ in (OH^-) . The resultant separation factors were in the range of 20-50.

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Metallex

Magnesium added to amalgams after reduction, but before washing, increases reduction yields, but increases the porosity and oxide content of the sintered metal.

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1.0 MASS-233 STUDIES (R. H. Rainey)

1.1 Thorex Second Cycle (R. G. Mansfield, A. B. Meservey)

Three different second cycle Thorex flowsheets gave a decontamination factor of 10 or less for processing the pilot plant first cycle product from the short-cooled material. The thorium and uranium were processed through the AC-ABC flowsheet and the BTC by both the acid and neutral flowsheet. In all three experiments the ruthenium (which constituted about 90% of the activity present) was in an extractable form and refluxed in the extraction column. After 3 volume changes there was about four times as much activity at the feed plate as was introduced in a single feed addition. The decontamination factor was 10 or less in all runs. The uranium and thorium losses were normal.

1.2 Organic Treatment (J. C. Ganchoff)

During the course of the short-cooled pilot plant run, the solvent increased in activity from the normal value of about 10^3 γ c/m/ml to about 3×10^6 γ c/m/ml. This activity was not removed by carbonate, lime, charcoal, silica gel, or any other method which has previously shown the ability to strip the activity from used solvent. There are indications that the activity is due to I^{131} . If the iodine has reacted with unsaturated organic material, it will be difficult to remove by ordinary cleanup methods.

1.3 Protactinium Recovery (A. B. Meservey)

Both the iodate and sodium hydroxide precipitation methods were successful in concentrating the protactinium from short-cooled Thorex A-column aqueous waste. Pa losses were 0.16% and 0.04% in the iodate

and hydroxide filtrates, respectively. Again, as in previous experiments, the iodate carried less fission product activity than the hydroxide.

2.0 VOLATILITY STUDIES (G. I. Cathers)

2.1 $\text{UF}_6 \cdot 3\text{NaF}$ Complex (R. L. Jolley)

The dependence of the rate of $\text{UF}_6 \cdot 3\text{NaF}$ complex decomposition on temperature is represented by the expression

$$\log r = 6.09 - 5.22 \times 10^3/T$$

where r is the rate in reciprocal minutes and T the absolute temperature. This equation is based on the data secured in fourteen runs over the temperature range of 245-355°C. The rate is defined as the fraction of $\text{UF}_6 \cdot 3\text{NaF}$ complex decomposed per minute. In order to calculate the results in this manner the UF_6 capacity on Harshaw NaF was assumed to be 1.33 g UF_6 per gram NaF. The temperature-dependence equation should therefore be valid for many types of NaF which differ in average crystallite size or UF_6 capacity.

3.0 THOREX PROCESS - SECOND URANIUM CYCLE (J. R. Flanary, J. H. Goode)

A total of four Thorex second uranium cycle solvent extraction runs, comprising 2925 grams of U^{233} , were made, completing the Chemical Section participation in the Pilot Plant Section's Thorex program. A total of twenty runs were made during the past three months, in which

nearly 17 kilograms of U^{233} were processed and returned to the Pilot Plant for evaporation and shipment. The uranium product met both fission product and ionic specifications in nearly all cases. Two bottles of product that did not meet fission product specifications resulted from a test run using a new diluent. This material was reprocessed and shipped. Essentially no losses of uranium occurred during the processing, and the material balances over the 17 kg of U^{233} were within the analytical error.

4.0 EQUIPMENT DECONTAMINATION (R. E. Leuze, R. D. Baybarz)

4.1 Preparation of Chromous Sulfate (R. L. Reese)

Electrolytic reduction of solutions containing 0.4 mole of either $Cr_2(SO_4)_3 \cdot K_2SO_4 \cdot 24H_2O$ or CrO_3 per liter of 1 M H_2SO_4 yielded solutions containing 97.5% or 94.5% chromous. In both processes amalgamated lead electrodes and an anion exchange diaphragm was employed. A current density of 0.10 amp/cm² for 1 hr was used to reduce the chromium potassium sulfate solution. A current density of 0.25 amp/cm² for 65 min was used to reduce the chromic acid to $Cr_2(SO_4)_3$. At this time the characteristic dark green color of $Cr_2(SO_4)_3$ appeared, and the current density was decreased to 0.10 amp/cm². Reduction at this current density was continued for 90 min.

Various other electrode combinations and cell designs were not satisfactory. The following cathode-anode pairs were used in a cell without a diaphragm: black iron-black iron, lead-black iron, and nickel-black iron. During the reduction, oxygen and $K_2Cr_2O_7$ were produced at the anode. Without a diaphragm to keep the anodic and cathodic solutions separated, any chromous ion formed was reoxidized.

5.0 HOMOGENEOUS REACTOR BLANKET PROCESSING (R. E. Leuze)

5.1 Plutonium Hydrolysis and Precipitation (J. M. Chilton)

There was no PuO_2 precipitate formed by heating 1.4 g UO_2SO_4 in a titanium-lined autoclave at 250°C after any of four daily additions of plutonium which gradually raised the plutonium concentration to 21.3 mg/kg H_2O . This is in agreement with previously reported results. The daily additions of about 5 mg Pu/kg H_2O /day will be continued to determine the concentration at which PuO_2 first forms and to determine the effect that the presence of PuO_2 has upon further precipitation. A titanium metal coupon in this solution adsorbed $0.08 \mu\text{g Pu/cm}^2$ during the heating time of approximately 100 hr.

The ineffectiveness of ZrO_2 as a carrier for plutonium from 1.4 g UO_2SO_4 at 250°C was shown. A repeat run of the experiment in which zirconium and plutonium were hydrolyzed and precipitated at 250°C from 1.4 g UO_2SO_4 again resulted in only partial precipitation of the plutonium. Solutions which originally contained 13.8, 23.1, and 46.4 mg Pu/kg H_2O analyzed 10.9, 18.9, and 39.0 mg/kg H_2O , respectively, after heating 60 hr at 250°C under 100 psi O_2 and 200 psi H_2 . The original zirconium concentration was about 100 mg/kg H_2O for all three solutions, and ZrO_2 was formed in all three solutions upon heating. The plutonium behavior in these experiments was similar to behavior in the absence of zirconium.

5.2 HRT Solids Dissolution (R. D. Baybarz)

Simulated HRT core solids containing ~60% ZrO_2 were more readily dissolved in H_3PO_4 after baking at 350°C for 15 min.

Simulated HRT core solids containing ~60% ZrO_2 were prepared by hydrolysis at 300°C . One gram of this material was dried and heated

at 350°C for about 15 min. This baked oxide was then dissolved by adding 20 ml of 85% H_3PO_4 and heating to remove water. Most of the solids were dissolved by the time the temperature reached 190°C, and dissolution was complete at 210°C. One gram of unbaked solids was treated in a similar manner. However, no dissolution was noticed until the temperature reached 225°C. Dissolution was complete at 240°C.

Attempts to prevent zirconium hydrolysis in 4 M H_3PO_4 were only partly successful. By saturating the solution with $Na_4P_2O_7$, most of the zirconium was held in solution; however, some hydrolysis was obtained by boiling (~100°C) for 24 hr. Partial zirconium holding was also obtained using a saturated $Al_2(SO_4)_3$ solution or by adding Thoron (R2-arsonic acid).

Scouting corrosion studies indicate that tantalum is suitable for containing phosphoric acid at 250°C. Corrosion rates of several metals are given in Table 5-1.

Table 5-1

Corrosion in Boiling Phosphoric Acid

Metal	Exposure Time (hr)	Exposure Temperature (°C)	Corrosion Rate (mpy)
Tantalum	48	250-260	0.67
Monel	20	235-250	110
Zircaloy-2	20	240-250	74
Hastalloy	16	240-250	390
347 Stainless Steel	16	240	4400
Titanium	1	230	170

6.0 HOMOGENEOUS REACTOR FUEL AND BLANKET STUDIES (J. P. McBride)

6.1 Thorium Oxide Slurry Irradiation Studies (W. A. Krohn)

LIR experiment No. 27 containing a 750 g Th/kg H₂O slurry of the standard oxide (#200-A-6) has been stirred in-pile for ten days at 300°C with no apparent change in slurry behavior. A control experiment has also operated in the laboratory for four days.

Viscosity calibration curves were prepared from data obtained with the dash-pot bomb using silicone oil and solenoid currents of 8, 9, 10, and 11 amps. Sensitivity of stirrer operation to viscosity changes decreased with increasing solenoid current.

Approximately 50 g of thorium--0.5% enriched uranium oxide were prepared by coprecipitating the oxalates and calcining them at 800°C. This material will be used in irradiation experiments for corrosion and fission product distribution data.

6.2 Development of Radiation Facilities (W. M. Woods)

Bomb dismantling equipment in Cell 7 of the HRLAF is in working order after correction of valve misassembly and numerous electrical faults and modification of the impact wrench. A wrench has been made which can be slipped over the bombs with the manipulators and permits a more positive gripping of the bomb by the vise on the bomb dismantling equipment. Manipulators are being installed. The cell will be strip-coated after a final check of the cell equipment with cold materials.

6.3 Characterization of Pumped Slurries (C. E. Schilling)

A 50 ml sample (as received) of the standard oxide was blended

for 3 hr at 26°C in the Waring blender. The resulting slurry had a very slow settling rate and a bulky sediment in contrast to the very rapid settling rate and dense sediment observed with the "as received" material. Pictures of the blended material showed that the spherical agglomerates (11 μ average size) of the standard oxide had been largely degraded into fines. Very few large fragments of the original spherical agglomerates were present.

A portion of the blended material was allowed to air-dry. The dried material was flaky and difficult to reslurry. Blending the suspension of the direct material for 1 hr resulted in an extremely slow settling slurry containing 80 g Th/kg H₂O.

6.4 Effect of Reagent Addition Rate on the Settling Rate of Thorium Oxalate (C. E. Schilling)

Large-scale preparation of 10°C precipitated oxalate has been slowed considerably by the fact that the precipitated material settles extremely slowly and must be washed by decantation. Laboratory studies showed that adding the thorium nitrate to the oxalic acid at a very slow rate accelerated the settling rate of the resulting oxalate precipitate.

Four oxalate precipitations were made by adding 1.0 M thorium nitrate solution to an 0.8 M oxalic acid solution (15% excess). With total addition times of 4.16, 26.8, 89.5, and 468 minutes the settling rates of the oxalate precipitate decreased from 1.21 to 1.85, 3.03, and 9.66 inches per hour. It is estimated that by increasing the reagent addition time in the pilot plant from 1 1/2 hr to 6 1/2 hr, the time required to wash the precipitate (5 washes) would be decreased by 2/3 (100 hr to 30 hr settling time).

7.0 HOMOGENEOUS REACTOR CHEMICAL PROCESSING (R. A. McNees)

Treatment of simulated HRT chemical plant solids with NaOH and Na_2CO_3 solutions at 250°C for 16 hr did not convert the solids into a form which could be dissolved in dilute H_2SO_4 or HNO_3 . The reaction vessels were stainless steel tubes closed with Parker fittings and were heated in a one-liter autoclave. One gram of solids with 5 ml of 10, 20, or 50% NaOH or saturated (at room temperature) Na_2CO_3 solutions were used. Corrosion was noticeable in all cases and the tube containing the 50% NaOH solution was corroded through at one spot. After the caustic digestion the solids were treated with dilute boiling solutions of HNO_3 and H_2SO_4 .

Dissolutions were 14.7% for the 10% NaOH; 9.0% for the 20% NaOH, 8.3% for the Na_2CO_3 solution. Part of the solids from the 50% NaOH treatment were lost due to tube failure.

Separate portions of three different solids were treated with two different mixtures of HCl and HNO_3 . One mixture was 3 volumes concentrated HCl and one volume concentrated HNO_3 ; the other was 2 N HNO_3 and 6 N HCl. Five grams of solids were refluxed with 50 ml of mixed acid for 16 hr. The solids remaining were weighed and analyzed for uranium. Results are shown in Table 7-1.

Table 7-1

Solid	Acid	Dissolved (%)	U(Original) (%)	U(Residue) (%)
A	Conc.	35.7	0.76	0.933
A	Dil.	37.2	0.76	0.834
B	Conc.	29.0	0.787	0.345
B	Dil.	32.2	0.787	0.358
C	Conc.	26.6	1.18	1.19
C	Dil.	27.6	1.18	1.12

From the data in Table 7-1 it is evident that mixtures of HCl and HNO_3 cannot be relied upon to either dissolve reactor solids or effectively leach uranium from them.

The uranium content of these samples of solids is higher than the 0.2% reported previously for solids obtained by direct hydrolysis at elevated temperature from 5 g U/kg H_2O - UO_2SO_4 solution. The high uranium content of sample A is due to the method of preparation of the solids. Precipitated hydroxides of iron, chromium, zirconium, and rare earths were treated at 300°C with UO_2SO_4 solution. Some of the uranium in the solution was adsorbed on the surfaces of the hydroxides and fixed there during the heating cycle. Sample B is solids from a Y-12 loop after several thousand hours of operation with concentrated, 1 M, UO_2SO_4 solutions. Sample C is from a Unit Operations loop run. The higher uranium content of sample C is a result of the precipitation of part of the uranium in the solution during operation of the loop.

7.2 Iodine Chemistry (S. Peterson)

The ratio of elemental to oxidized iodine in simulated homogeneous reactor fuel solution (0.02 M UO_2SO_4 , 0.005 M H_2SO_4 , 0.005 M CuSO_4 , 1×10^{-5} M HIO_3 , 200 psi O_2 at 25°C) at 100°C ranged from 0.5/1 to 1.2/1 in the absence of radiation. In a gamma flux of 8.1×10^{17} ev/g $\text{H}_2\text{O}/\text{min}$ and at a temperature of 100°C the ratio of elemental to oxidized iodine dropped to the range of 0.03/1 to 0.02/1. In another experiment the Co^{60} source was lowered around the reaction vessel during the experiment. With the source out of position the ratio was 0.5/1; with the source in position, the ratio dropped to 0.02/1. It thus becomes apparent that the effect of gamma radiation on the valence distribution of iodine at 100°C is opposite to what it is at 250°C .

8.0 HEAD-END PROCESSING (J. E. Savolainen)

8.1 Dissolution Studies (A. H. Kibbey)

The desirability of dejacketing fuels clad with a thin layer of Zr or Zircaloy-2 prior to core dissolution in aqueous nitric acid or aqua regia prompted a study of the dissolution rates of these materials in 1 M HF, 1 M HF-1 M HNO₃, and 1 M HF-1 M HNO₃-1 M HCl solutions at room temperature and at -90°C. The results of these dissolution studies are summarized in Table 8-1. It should be noted that a Zr dissolution rate of 1 mg/min/cm² corresponds to a dejacketing time of 17 min/mil of Zr cladding.

Table 8-1

Material	Temperature (°C)	Dissolution Rates (mg/min/cm ²)		
		1 M HF	1 M HF-1 M HNO ₃	1 M HF-1 M HNO ₃ -1 M HCl
Zr (APDA)	25	0.22*	0.20	---
	90	11.8	15.8	---
Zircaloy-2	25	0.22	0.27	0.326
	90	5.37	6.96	2.05
Ti	25	0.037	0.078	---
	90	1.50	2.38	---
Ta	25	---	---	$< 1 \times 10^{-5}$
	90	---	---	2.04×10^{-2}

* At 100% excess U loss was 0.42%.

8.2 Zircex (J. E. Savolainen)

Two experiments were made to evaluate the removal of Zircaloy-2 cladding by the Zircex process from UO₂ core material. Sections of a

PWR UO_2 core fuel rod were hydrochlorinated at 300°C . Some chloride pickup by the UO_2 was observed, presumably forming UO_2Cl_2 . A small fraction, less than 1% of the uranium dioxide, was not soluble in boiling 2 M HNO_3 . This insolubility is not expected to have been caused by the hydrochlorination step. The results are listed in Table 8-2.

Table 8-2

Hydrochlorination Time (min)	Cladding Removed (%)	UO_2 found as UO_2Cl_2 (%)	UO_2 Insoluble in 2 M HNO_3 (%)
56	86	2.9	0.331
120	100	5.38	0.44

The overall material balances were 97.3 and 97.5%.

9.0 PHYSICAL STUDIES (R. G. Wyner)

9.1 Mice (L. M. Ferris)

It was previously reported that the U resulting from vaporization of the metal under water is non-ionic and probably exists as UO_2 . Acidification of the solution to render the U ionic is not economically attractive in the Mice scheme. On the scale expected for the Mice scheme it would cost on the order of $\$30 \times 10^6$ to make the solution 10^{-2} M in HNO_3 . It was determined earlier that this concentration of HNO_3 is not nearly sufficient to readily convert the suspended U to an ionic form. A better approach appears to be concentration of the U by

scavenging. A series of experiments were performed to determine whether hydrous ferric oxide could be used as a scavenging agent. No precipitate was formed in solutions containing 50-500 mg Fe/liter added as $\text{Fe}(\text{NO}_3)_3$ even after refluxing for several hours. Coloration of the solutions and pH measurements indicate that the Fe is hydrolyzed and probably exists as a stable sol. Precipitates of hydrous ferric oxide were produced in a solution containing 50 mg Fe/liter by adjusting the hydroxyl ion concentration to $\sim 3 \times 10^{-3} \text{ M}$. This concentration of base did not cause precipitation in solutions where the Fe concentration was greater than 50 mg/liter. Also hydroxyl ion concentrations in the range of $\sim 1.5 \times 10^{-2} \text{ M}$ to 1.8 M apparently stabilize the 50 mg/liter Fe sols. Separation factors of the order of 20-50 were obtained by scavenging U in a concentration of $\sim 0.1 \text{ mg/liter}$ in solutions containing 50 mg Fe/liter and $\sim 3 \times 10^{-3} \text{ M}$ base. The cost of the amount of NH_3 needed would be of the order of $\$0.5 \times 10^6$.

10.0 MERCURY PROCESSING STUDIES (O. C. Dean)

10.1 Metallex (O. C. Dean)

Efforts to evaluate the effects of magnesium additions to Metallex amalgams were continued. A summary of results to date are given in Table 10-1. If the magnesium is added subsequent to the reduction step, but prior to washing, it appears to increase the reduction yield significantly. Addition of magnesium prior to reduction has only a slight effect, if any, on increasing reduction yields. Free voidage (atmosphere-connected porosity) and oxide content of the sintered metal appear to be increased by use of

magnesium. The high residual magnesium in the sinters point to the necessity for increasing the acid concentration in the aqueous amalgam wash, which is 1.0 M HCl at present. The high magnesium residue may account for increased porosity and oxide. Further evaluation is in progress.

Table 10-1

Reduction Run	Reduction Yield (%)	Thorium Recovery in Quasi (%)	Free Voidage on Sinter (%)	Mg in Sinter (ppm)	HCl Insoluble in Sinter (%)
Average run, no Mg	65-75	50-60	7-25	---	0.5-2.0
Average run, 0.03 wt % Mg added after reduction	89.4	89.0	19 ^b	1200	3.0
Average run, 0.003 wt % Mg added before reduction	75.7	72.8 ^a	24.4	545	3.7
Average run, 0.03 wt % Mg added before reduction	81.0	57.0	---	---	---

^a 37% of the thorium recovered in immediate amalgam filtration, and the rest recovered by a second filtration 16 hr later.

^b Average of two values.

R. E. Blanco

D. E. Ferguson

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