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D. E. Ferguson

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SUMMARY

Homogeneous Reactor Fuel Processing

At 250°C the presence of small amounts of hydrogen (5-10 psi H₂, 200 psi O₂ at 25°C) increases the ratio of free to combined iodine in dilute uranyl sulfate solution. Larger amounts of hydrogen (50-55 psi H₂, 200 psi O₂) apparently decrease this ratio below what it is with no hydrogen present.

Eleven molar sulfuric acid continues to be the most promising solvent for simulated homogeneous reactor corrosion and fission product solids.

Uranium-233 Isolation

Addition of HNO₃ to the acetate products reduces the amount of uranium precipitation but does not completely eliminate it.

When 5 N HF was used as an elutriant, 93% of the uranium was removed from Dowex 50W resin in two column volumes, and the uranium concentration was 220 g/l.

Volatility Studies

A sludge consisting of 37.5% NiF₂ was found in the waste NaF-ZrF₄ salt used in the fluorination tests with UO₂F₂. Vapor pressure data obtained for UF₆ over 3KF·2UF₆ was erratic due to the low capacity of KF for UF₆ and possibly, the low porosity of the KF.

Mass-233 Studies

The decontamination factor for thorium is increased by a factor of

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six by the addition of 0.003 M H_3PO_4 to the second cycle scrub solution. No effect was noted from the use of 0.005 M $\text{H}_2\text{C}_2\text{O}_4$. Recycle of second cycle aqueous waste as first cycle scrub gave satisfactory operation. A fission product decontamination factor of 10^5 was obtained for 97% of the uranium sorbed on a cation resin column by elution with acidified TBP. No difficulty was encountered in making the transition between aqueous and organic phases.

Heterogeneous Power Reactor Processing

A number of alloys of uranium with Nb, Zr, Mo, and Si were readily dissolved in 2 M HCl--5 M HNO_3 . The maximum loss of uranium in undissolved solids was 1.4% in the case of 4.5% Zr--0.5% Nb--95% U alloy.

Fused Salt Processing

A maximum STR element dissolution rate of 1.0 mg/min/cm² was obtained in a series of three experiments at 600°C by bubbling HF into a NaF to ZrF_4 1:1 mole ratio melt.

Excer

A tap density of 3.5 was obtained on $\text{UF}_4 \cdot \frac{3}{4}\text{H}_2\text{O}$ when 0.5 M HCl was the electrolyte compared to 2.5--2.7 when sulfuric acid was the electrolyte.

Waste Studies

Strontium and cesium decontamination factors of 17 and 18 were obtained in initial Diban runs using the 1-in.-diam continuous column.

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Fluorox

A complete cycle of reduction, hydrofluorination, and oxidation was completed on African ore concentrate and the product submitted for impurity analysis.

North Carolina State Reactor Fuel Recovery

A total of 871 g of enriched uranium was recovered from about 13 liters of uranyl sulfate solution from the North Carolina State College reactor.

Feed Materials

Hermex

Forty-five day-cooled, 2×10^{17} nvt uranium was dissolved in boiling Hg at the rate of $12 \text{ mg/cm}^2/\text{min}$. Gross β and gross γ decontamination of the sample was 15 and 28 respectively. Sr and TRE activities were efficiently removed, while Zr, Nb and Ru removal was poor.

Metallex

A Disperser agitator with squirrel cage baffle in the Mark II mixer gave approximately the same performance results as the turbo mixers.

All of the iron, and 52% of the nickel present in the anhydrous ThCl_4 feed salt carried over into the final metal product.

Non-uniform, but adherent deposits of a metal which is chiefly titanium were formed on Zircaloy-2 which had been coated with zinc and titanium quasi-amalgam and heated.

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1.0 HOMOGENEOUS REACTOR FUEL PROCESSING
(R. A. McNees)

1.1 Iodine Chemistry (S. Peterson)

In simulated fuel solution at 250°C under 200 psi of oxygen pressure (measured at 26°C) after 2 hr heating the ratio of free to combined iodine is fairly constant in the range of 10 ± 2 . With 5-15 psi of hydrogen in addition to 200 psi of oxygen the ratio was more erratic, but was usually larger and ranged between seven and twenty. With more hydrogen (49-55 psi) the ratio was erratic but usually lower and ranged between two and eleven. The presence of copper in the solution (0.005 $\underline{m}$ CuSO_4) had no visible effect other than catalyzing the combination of the gases.

1.2 Solids Dissolution (R. A. McNees)

Four new batches of solids have been obtained from Y-12 loops and tested for their dissolution characteristics. Under identical volume, weight conditions (30 ml of dissolver solution to 100 mg of solids) dissolutions ranging from 30 to 80% for different solids were obtained with 0.35 $\underline{N}$ CrSO_4 , 1.0 $\underline{m}$ H_2SO_4 solutions at 75-80°C. Nitric acid solutions ranging from 15 $\underline{M}$ to 3.5 $\underline{M}$ were equally ineffective. At 165-170°C, 10.8 $\underline{M}$ H_2SO_4 gave dissolutions ranging from 93 to 99+%.

A synthetic solid containing 90% Zircaloy-2 corrosion products, 5% stainless steel corrosion products and 5% fission product solids was prepared and aged for 1 week at 300°C. Under the same conditions as given above 10.8 $\underline{M}$ H_2SO_4 gave 98% dissolution while CrSO_4 solution gave 6% dissolution. Treatment of 1.002 g of this solid with three successive 10 ml portions of 10.8 $\underline{M}$ H_2SO_4 gave a cumulative dissolution of 50.88 and 98%. Although not yet analyzed, the residue appears to be largely ruthenium.

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2.0 URANIUM-233 ISOLATION (W. E. Tomlin)

Since the present isolation process used in the Thorex pilot plant is not satisfactory, alternate isolation methods are being investigated. The principal difficulties with the present process are uranium precipitation in the product, interference of acetate and citrate when recycled through solvent extraction, and the careful control required during elution. In addition to these, it is desirable to increase decontamination so thorium with long irradiation and short cooling may be satisfactorily processed with only one solvent extraction cycle for uranium.

Addition of nitric acid to the acetate product reduces the amount of precipitation but does not completely eliminate it. Nitric acid is added to products from the present production runs in order to allow sampling before any appreciable precipitation.

When 5 N HF was tested as an alternate elutriant 93% of the uranium was eluted in 2 column volumes, and the uranium concentration was 220 g/l. Elution with 1 N HF removed 81% of the uranium in 4.4 column volumes, and elution with 2 N HF removed 89% of the uranium in 3.3 column volumes.

3.0 VOLATILITY STUDIES (G. I. Cathers)

3.1 Fused Salt-Fluorination Tests (M. R. Bennett)

The formation of a nickel salt sludge was observed in one batch of NaF-ZrF_4 salt after several fused salt fluorination tests. This sludge was detected as a thick and putty-like material at the bottom of the reactor, and amounted to 13.7 g or 3.7% of the total charge of

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salt of 375 g. The sludge was isolated by decantation of the molten supernatant salt. The nickel content of the sludge was 22.8%, corresponding to 37.5% as NiF_2 . The Zr/Na mole ratio in the sludge was 0.83 in comparison to 0.95 in the supernate. This difference is probably not significant, and indicates that neither NaF or ZrF_4 precipitates preferentially with the NiF_2 as in the case of CrF_2 . The nickel content in the supernatant salt was 0.10%. The batch of salt in which the sludge was detected had an unusual history in that UO_2F_2 had been added and UF_6 fluorinated out of the salt in two successive runs.

3.2 UF_6 Complex Studies (R. L. Jolley)

Study of the vapor pressure of UF_6 over the UF_6 -KF complex by the transpiration method has given erratic results due to the seeming lack of porosity of the Harshaw KF pellets employed, and, possibly, to a more complicated chemical equilibrium than in the case of the UF_6 -NaF complex. In one test with 12-20 mesh material obtained from Harshaw KF pellets, the capacity appeared to be 0.184 g U per gram salt, far below the theoretical value of 2.74 for the case of the $2\text{UF}_6 \cdot 3\text{KF}$ complex reported in the literature. The erratic vapor pressure measurements probably resulted from the low capacity, and the fact that the absorbed UF_6 appeared to be only on the outer surface. The lack of UF_6 penetration into the KF granules was evident in aqueous dissolution of the salt. Further study of the UF_6 -KF system has been suspended until a more suitable form of KF has been obtained.

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

4.1 Thorex Second Cycle Development (R. G. Mansfield)

A series of D-column countercurrent experiments were run in order to compare the decontamination factors obtained under varying conditions. Due to the low level of activity in the feed, decontamination factors cannot be determined accurately but a comparison of the effect of scrub additives was obtained.

	Decontamination Factor				
	Gross γ	Pa γ	Zr-Nb γ	Ru γ	TRE β
No additive	25	15	50	50	10^3
0.003 M PO_4	150	>50	200	5	90
0.005 M $\text{H}_2\text{C}_2\text{O}_4$	20	5	40	4	200

No operational difficulty was found from the recycle of aqueous waste stream from the D-column as first cycle scrub solution.

The use of 0.5 M $\text{Al}(\text{NO}_3)_3$ instead of 0.35 M $\text{Al}(\text{NO}_3)_3$ as the D-column scrub did not decrease the thorium loss or give other apparent advantage.

4.2 Recovery of Traces of U^{233} from Solvent Wash Solution (A. B. Meservey)

From time to time there has been as much as 1% loss of U^{233} from the Thorex plant in the solvent treatment system. Methods for the recovery of this material have been investigated at the request of the Pilot Plant. A sample of the neutralized and concentrated solvent wash system waste (TWC) was supplied for investigation which contained $3.08 \times 10^3 \alpha \text{ c/m/ml}$. In two extraction passes with equal volumes of aqueous and 42.5% TBP, the solution was reduced to essentially zero

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activity. Since this ease of extraction is contrary to previous experience, it seems that the sample supplied is not typical of the material previously reported to be difficult to extract.

4.3 Elution of Uranium from Resin Column with TBP (A. B. Meservey)

In confirmation of preliminary earlier work, the elution of uranium from an ion exchange resin column with TBP has given high fission product decontamination. Approximately 97% of the uranium was eluted in 2.3 volume changes with a fission product activity decontamination factor of about 10^5 . The remaining 3% in 1.7 volume changes had a decontamination factor of over 300. No difficulty was encountered in making the transitions between aqueous and organic phases. The present elution system using acetate-citrate gives essentially no decontamination.

5.0 HETEROGENEOUS POWER REACTOR PROCESSING (J. E. Savolainen)

5.1 Dissolution of Uranium Alloys in Aqua Regia (A. H. Kibbey)

Various Nb, Zr, Mo, and Si alloys of uranium were dissolved in 2 M HCl--5 M HNO₃ solution. The insoluble residues were removed and washed by centrifugation. The supernates and solid residues were analyzed for uranium. Results of these experimental dissolutions are summarized in Table 1. The maximum U loss was 1.4% in the case of 4.5% Zr-0.5% Nb-95% U alloy.

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Table 1
Dissolution of Various Uranium Alloys in Aqua Regia

Alloy Composition - %					% of Uranium Retained by Residue
U	Nb	Zr	Mo	Si	
98	2	--	--	--	0.16
95	5	--	--	--	0.98
90.3	9.7	--	--	--	0.05
85	15	--	--	--	0.57
95	0.5	4.5	--	--	1.40
93.4	1.33	5.27	--	--	1.07
99.25	0.12	0.41	0.22	--	0.02
98	--	--	2	--	0.00 (complete dissolution)
99.5	--	--	--	0.5	0.002

6.0 FUSED SALT PROCESSING (R. G. Wymer)

A maximum STR element dissolution rate of 1.0 mg/min/cm^2 was obtained in a series of three experiments at 600°C by bubbling HF into a NaF to ZrF_4 1:1 mole ratio melt.

The product (percent HF utilization) x (rate of STR dissolution in $\text{mg/min}\cdot\text{cm}^2$) has a constant value of ~ 42 as determined both from the above experiments, in which HF was bubbled into 100 ml of molten salt at rates of 25-83 mg/min, and from Argonne experiments, in which HF was bubbled into 50 ml of molten salt at rates of $\sim 600 \text{ mg/min}$.

7.0 EXCER
(I. R. Higgins)

Several flowsheet variations are considered for purification of uranium concentrates prior to the electrolytic reduction to $\text{UF}_4 \cdot \frac{3}{4} \text{H}_2\text{O}$. A sample of Lukachukai diuranate, obtained from earlier studies with ore pulp on the 2-in. contactor, was dissolved in 8 M HCl, sorbed on an anion resin, eluted with water as UO_2Cl_2 , and reduced in the electrolytic cell. The final dehydrate UF_4 analyzed 4 ppm in Fe, or a factor of 15 below specifications. No decontamination was obtained in the HCl-anion step but the electrolytic cell contributed a D.F. of 2300. A sample of Utex diuranate was dissolved in H_2SO_4 and sorbed on an anion column. The SO_4 was removed with 8 M HCl and the U eluted with water. The UO_2Cl_2 was converted to UO_2F_2 on a cation column and reduced in the electrolytic cell. The final UF_4 analyzed 10 ppm in Fe. The anion column contributed a D.F. of 11, the cation none, and the electrolysis 250. Neither of the final products were analyzed for the total contaminants because each indicated a Ca content greater than 100 ppm.

A simulated impure UO_2Cl_2 was converted to UO_2F_2 and the ionized cations removed on a second cation column. Sodium removal was very effective with a D.F. of 1300, but Ca was only 4.

The factors which influence product density have been very evasive, but the problem has apparently been solved. When 0.5 M HCl was used to carry the current instead of H_2SO_4 , the $\text{UF}_4 \cdot \frac{3}{4} \text{H}_2\text{O}$ tap density was 3.5 instead of 2.5--2.7.

8.0 WASTE STUDIES
(I. R. Higgins)

Two diban - 1 inch contactor runs were made, one with Cs^{137} tracer

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and the other with Sr^{90} . The feed rate was reduced approximately one-half from a run which gave a D.F. of 220 for Cs. The average Cs D.F. was 17 with a maximum of 100. The decontamination was continuously improving which indicated steady state had not been attained. The one Sr D.F. was 18 and was apparently steady. The hope was to squeeze in some high decontamination demonstrations, but obviously some more thorough investigations are required.

9.0 FLUOROX (R. G. Wymer)

9.1 Ore Concentrate Studies (L. M. Ferris)

To determine the extent of purification which might be effected by each stage of the Fluorox process, about 10 g of African ore concentrate pellets were simultaneously starch reduced and hydrofluorinated at temperatures from 500 to 700°C. The impure UF_4 product was then oxidized to UO_2F_2 and UF_6 with dry O_2 at 800°C. The results of the chemical and spectrographic analyses of the respective products are not yet available. In the reduction-hydrofluorination step CaSiF_6 and $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ (positively identified by x-ray analysis) were found at the cool exit-end of the reaction tube.

10.0 NORTH CAROLINA STATE REACTOR FUEL RECOVERY (J. R. Flanary, J. H. Goode)

A total of 871 grams of enriched uranium was recovered from about 13 liters of uranyl sulfate solution from the North Carolina State reactor. The fuel was made 1.8 M in aluminum nitrate and 1 M in nitric acid by the addition of crystalline aluminum nitrate and nitric

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acid, and processed through two columns under the following conditions:

AF/30 ml per min: ~ 40 g U/l, 1.8 M Al, 1 M HNO_3 , ~ 0.17 M $\text{SO}_4^{=}$

AS/6 ml per min: 0.75 M Al, 1 M HNO_3 , 0.01 M $\text{Fe}(\text{NH}_2\text{SO}_3)_2$

AX/30 ml per min: 15% TBP in Amsco 125-82

BX/30 ml per min: 0.01 M HNO_3

Total process uranium losses were 0.008%. The gross β decontamination factor was estimated at 1×10^5 and the gross γ D.F. was 2.4×10^3 . Other fission product D.F.'s were Ru, 4.6×10^3 ; Zr-Nb, 2.3×10^3 ; and TRE, 2.3×10^5 .

11.0 FEED MATERIALS (O. C. Dean)

11.1 Hermex (B. H. Morrison)

11.1.1 Decontamination of Short Cooled Irradiated Uranium

A sample of 45-day-cooled, irradiated (nvt, 2×10^{17}) uranium was decontaminated by the Hermex process to the extent of 28 for gross β and 15 for gross γ . The irradiated uranium dissolved at a rate of 12 mg/cm²/min in boiling (356°C) mercury.

A 15 g section of natural uranium was irradiated for 63 hr at an average neutron flux of 8.3×10^{11} n/cm²/sec in the graphite pile. After cooling for 45 days the uranium was dissolved at a rate of 12 mg/cm²/min in the laboratory continuous dissolver by treatment with four successive 220 ml portions of mercury at 356°C. The $\sim 0.2\%$ uranium amalgam was drained from the dissolver, cooled to 25°C, and concentrated to $\sim 2\%$ uranium by vacuum filtration. The $\sim 2\%$ uranium quasi-amalgam was reslurried with twice its volume of clean mercury and washed eight times with 100 ml portions of 1 N HCl. The excess mercury was removed from dilute amalgam by vacuum filtration and the

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approximately 3% uranium quasi-amalgam charged to the furnace. Slow heating to 1300°C volatilized off the mercury and melted the uranium. The overall yield from metal to metal was 71%.

The specific activities and decontamination factors for the starting and product uranium are listed in Table 2.

Table 2

Decontamination Factors for Short-Cooled Uranium

$nvt = 2 \times 10^7$, 45 day cooled

Specific Activity	Starting U c/m/g U	Product U c/m/g U	Decontamination Factor
Gross γ	7.8×10^8	5.4×10^7	15
Gross β	2.0×10^8	6.9×10^6	28
Cs β	4.1×10^5	2.6×10^4	16
Sr β	2.4×10^7	3.8×10^4	1450
TRE β	9.5×10^7	1.5×10^5	650
Ru β	2.7×10^6	1.7×10^6	~ 2
Zr γ	7.8×10^7	8.5×10^6	~ 10
Nb γ	6.1×10^7	9.8×10^6	~ 6
Pu α	2.2×10^5	$3.4 \times 10^3(?)$	64

The gross D.F.'s obtained in this run are lower by a factor of ten than those observed with 3 1/2-year-cooled irradiated uranium. Sr and TRE β activities are efficiently removed and Cs β activity was low in the initial material. However, Ru, Zr, and Nb removal was poor. In future runs the uranium quasi-amalgam will be washed several times with clean mercury in hope that Ru, Zr, and Nb can be extracted in the mercury filtrate.

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11.2 Metallex (O. C. Dean)

11.2.1 Reduction of ThCl_4 - Evaluation of Disperserator Agitator

One hundred thirty grams of Fernald anhydrous thorium tetrachloride, whose analysis was 13.9% methanol-insoluble (86.1% pure ThCl_4) 0.22% H_2O , 36 ppm Fe, 444 ppm Ni and <10 ppm Cr were reduced in the Mark II high speed mixer with 400 ml of 3.5 M sodium amalgam at 130°C for one hour. The amount of sodium used was stoichiometric with respect to the total thorium in the run. The agitator was a double-coned Disperserator type, operating at 2000 rpm. The baffle was a squirrel-cage type spaced 1 in. from the agitator. This agitator and baffle system had not been used before, and one of the objectives of the run was to compare its effectiveness with the turbo-mixers used in previous runs. Sixty-seven percent of the total thorium and 77.4% of the available thorium (from the pure ThCl_4 content) were reduced to metal. Fifty-eight percent of the total thorium and 68% of the available thorium were recovered in the quasi-amalgam product. The thorium balance was 105% for the run. This was approximately the same yield as would be expected from previous equipment under similar conditions.

An attempt was made to estimate the thorium reduced from the initial and final sodium analyses of the unwashed amalgam. It had been established from previous runs that 99 to 100% of the NaCl was removed from the unwashed amalgam by filtration. Analysis of the filtered final amalgam from the above run showed less than 5 ppm Cl^- and 1.4 M Na. The sodium usage appeared to be 10% less than that necessary to reduce the amount of Th found in the final product. Basing yield calculations on sodium analyses would be economical and convenient but thus far has been shown inaccurate.

11.2.2 Decontamination Studies

The extent to which nickel, chromium and iron from feed salt

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contaminates the metal product was also studied in this run. Special attention was given to washing the amalgam with a mechanical stirrer under first 1 N HCl then deaerated water. The amalgam was agitated for ~ 2 hr with each solution. The quasi-amalgam product, and the final sintered metal product were analyzed for Fe, Cr, and Ni. The iron analysis of the quasi-amalgam was such that no conclusion could be drawn. One hundred fourteen percent of the iron in the feed salt carried through into the final metal. Eighty-two percent of the nickel present in the feed salt carried into the quasi-amalgam, and 52% was found in the final sintered metal. No results on Cr were available.

Mixing rate studies showed that ThCl_4 and sodium amalgam in the turbo-mixers used were not uniformly mixed with respect to the Th/Hg ratio until after 5 to 10 minutes mixing, depending on the impeller and baffle systems used. The kinetic data collected in all of the reduction runs was for mixing times greater than 5 minutes.

11.2.3 Titanium Deposits on Zircaloy-2 (C. P. Johnston)

In six experiments Zircaloy-2 was coated with zinc, wetted with mercury, and recoated with titanium quasi-amalgam. The mercury and zinc was then removed by either vacuum retorting or heating under a purified argon atmosphere.

Adherent but non-uniform metallic deposits on the Zircaloy specimen have been observed. Analytical results are not available, hence the composition of the deposits are not known at this time.

In another experiment the Zircaloy was coated with a very thin layer of tin and amalgamated with titanium quasi-amalgam. From the titanium-tin diagram a Ti-Sn solid solution is known to exist below 950°C for compositions up to 18% titanium. It is hoped that a titanium crystalline surface may be developed, bonded to the Zircaloy

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by an Sn-Ti solid solution. The above sample has not as yet been subjected to retort treatment.

R E Blanco

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D E Ferguson

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