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CHEMICAL TECHNOLOGY DIVISION

MONTHLY REPORT FOR JULY 1963

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## ABSTRACT

Process development studies are reported on Power Reactor Fuel Processing (Shear and Leach Studies, Aqueous Process Development of Uranium Carbide Reactor Fuel, Corrosion Studies, Fluoride Volatility Processing: Aluminum Alloy Flowsheet Development, Fluoride Volatility Processing: NaF Sorption Studies, Fluoride Volatility Processing: Corrosion, and Fluoride Volatility Pilot Plant); Chemistry Research (Separations Chemistry Research, Separations Process Development, and Volatility Studies); and Peaceful Uses of Nuclear Explosions (Chemical Applications of Nuclear Explosions).

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## POWER REACTOR FUEL PROCESSING

Shear and Leach Studies. -- The engineering-scale development of the reactor-fuel shear and leach equipment, consisting of a 250-ton prototype shear, conveyor-feeder, and leacher, was continued.

Particle-size measurements were made on sheared sections of stainless-steel-clad  $UO_2$ ,  $UO_2-ThO_2$ , and Zircaloy-2-clad  $UO_2$ . The sheared sections were 1, 1-1/2, and 2 in. long. The amount of core material dislodged from Zircaloy-2-clad  $UO_2$ , sheared into 2-in. lengths was 73%, a factor of 5.8 and 4.2 more than stainless-steel-clad  $UO_2$  and  $UO_2-ThO_2$ , respectively.

Batch leaching of stainless-steel-clad oxide reactor fuels sheared to 1/2- to 2-in. lengths was also studied. In these tests the pieces were placed in a perforated basket which was then suspended in a batch dissolver containing recirculating acid for leaching the  $ThO_2-UO_2$  or  $UO_2$ . Factors studied included sheared length, basket-perforation size, and air sparge rates for the suspension of fines.

Batch leaching of 1-in. sheared lengths of stainless-steel-clad  $ThO_2-UO_2$  contained in a slotted basket (0.015-in.-side slots, 23% open area, ratio of the diameter of the basket to the diameter of the tank = 0.81) and air sparged at less than 1.2 scfm/ft<sup>2</sup> resulted in the most rapid leaching observed to date. Thorium dissolution was 90% complete in 2.9 hr and 99% complete in 7.4. A similar run, identical except for the omission of air sparging, required 5.7 hr to achieve 90% dissolution and about 13 hr for 99% dissolution.

Packing-density measurements on sheared stainless-steel-clad  $ThO_2-UO_2$  in 3- to 6-in.-diam cylinders were 4.4 to 5.1 g/cm<sup>3</sup> for 1/2-in. sheared lengths, and 3.7 and 3.9 g/cm<sup>3</sup> for 1- and 1-1/2-in. lengths. The uncertainty in this values may be as high as  $\pm 10\%$ .

With the stepped blade, a maximum force of about 156 tons was required to shear a bundle consisting of forty-nine 5/16-in.-OD solid 304 stainless steel rods.

Aqueous Process Development of Uranium Carbide Reactor Fuel. -- The irradiation of impure uranium monocarbide (5.0% C) or high-purity uranium

monocarbide (4.8% C) to 5000 to 7000 Mwd/ton had a significant effect on the chemical reactivity of this fuel material. In recent hot-cell studies, the irradiated specimens were almost nonreactive in water at 80 and 90°C, whereas unirradiated specimens of the same original composition were completely hydrolyzed to uranium dioxide in 4 to 6 hr under comparable conditions. Reactivity of the irradiated material in mineral acids, however, appears to be greatly accelerated. Dissolution of the water-soaked pellets in 6 M HCl at 80°C was completed in 5 min, compared with 6 hr for unirradiated specimens; gas evolution was too rapid for measurement. The reaction rate in 2 M HCl was about the same as that of 6 M at the unirradiated or 500-Mwd/ton level. Dissolution in 15.8 M HNO<sub>3</sub> was completed in less than 30 min, compared with the 2 hr required for unirradiated material.

Since the hydrolysis-dissolution head-end flowsheet developed for this fuel cannot be applied unless passivity to hydrolysis can be overcome, an alternative head-end flowsheet is being developed in which unirradiated uranium monocarbide is dissolved in excess 15.8 M HNO<sub>3</sub> in 2 hr, yielding a dark brown, solids-free solution containing 100 g of uranium per liter and 12.8 M HNO<sub>3</sub>. The partial destruction of organic acids is accomplished by 16-hr reflux of the strong nitric acid solution and subsequent distillation of the excess acid. After distillation of the excess acid, the uranyl nitrate concentrate is diluted with water to yield a stable raw feed solution containing about 375 g of uranium per liter and 1 M HNO<sub>3</sub>; the color is yellow-orange. The remaining organic impurities finally are destroyed by 2-hr reflux with 0.05 M Ce(NH<sub>4</sub>)<sub>2</sub>(NO<sub>3</sub>)<sub>6</sub>. Feed solution thus prepared performed adequately in batch extraction and stripping tests under simulated Purex solvent extraction conditions. Uranium recovery was quantitative.

Corrosion Studies. -- Nichrome V, A-nickel, and Haynes 25 alloy corroded at rates of 1.4, 1.2, and 31.1 mils/month during exposure to 13, 14, and 4 cyclic oxidation-chlorination tests, respectively. The cycle consisted in exposure of the alloy specimen, which was buried in graphite-UO<sub>2</sub> moistened with HNO<sub>3</sub>-HF, to (1) oxygen at 800°C for 5 hr and (2) exposure to chlorine at 500°C for 5 hr. In a similar cycle in which

the chlorine exposure was replaced by chlorination in refluxing hexachloropropene for 3 hr at 180°C, the Nichrome V and Haynes 25 showed slight weight gains over 12 and 3 cycles, respectively, while A-nickel and Carpenter 20 showed maximum corrosion rates of 8.0 and 1.2 mils/month over 11 and 9 cycles, respectively.

Hastelloy-B exposed for 304 hr in boiling 3 M  $ZrOCl_2$ --3 M HCl under an atmosphere of HCl- $H_2$  corroded at nearly constant rates of 0.8 mil/month in the vapor and 1.0 mil/month in the solution. No intergranular attack was noted. A specimen from a different heat, which corroded at about twice these rates and showed intergranular attack, was found to be highly contaminated with foreign material.

Fluoride Volatility Processing: Aluminum Alloy Flowsheet Development. -- At 600°C, the apparent maximum practical temperature for processing aluminum fuel elements, preliminary phase equilibrium data (ORNL-3462, p 9) indicated that KF-ZrF<sub>4</sub> melts had sufficient AlF<sub>3</sub> solubility to allow their use as the solvent in a molten salt volatility process for Al-U alloy fuels. Additional data have since been obtained, principally by a new visual method, which better defined the 600°C isotherm and confirmed the choice of an approximate composition path of the process. Purification of the components, KF, ZrF<sub>4</sub>, and AlF<sub>3</sub>, by volatilization was necessary to obtain melts that were sufficiently clear for visual study.

Mixtures of KF-LiF were also studied as potentially lower-priced Al-U alloy fuel solvents for the volatility process. Although the binary system has a low-melting eutectic, the liquidus temperature rose so steeply upon the addition of AlF<sub>3</sub> that only 3 mole % could be dissolved at 600°C. The minimum solidus temperature along the LiF-3KF·AlF<sub>3</sub> quasibinary section was found to be 720°C. There is a 490°C eutectic at the approximate KF-LiF-AlF<sub>3</sub> ternary composition (51-6-43 mole %), but melts in this region would have a capacity for at most about 6 mole % additional AlF<sub>3</sub> at 600°C.

Further laboratory studies of the KF-ZrF<sub>4</sub> salt system were made in order to determine aluminum dissolution rates and to more definitely establish the chemical reactions involved. Gas evolution tests showed that the dissolution of aluminum in molten KF-ZrF<sub>4</sub> at 600°C with hydrogen

fluoride proceeds with the liberation of only 25 to 35% of the theoretical amount of hydrogen. A digestion period during which the melt receives a continued hydrofluorination treatment results in the evolution of the remainder of the hydrogen and the elimination of a black residue in the salt. These observations reaffirm that only part of the aluminum dissolves through an acid-metal reaction; the remainder, and perhaps a major part, dissolves through the reduction (by the aluminum) of  $ZrF_4$  to form amorphous zirconium. Further use of the hydrogen evolution procedure is planned to establish the relative hydrogen fluoride and  $ZrF_4$  dissolution effects at various salt concentrations.

A modified Al-U alloy process flowsheet was devised to increase the process capacity, that is, the amount of fuel processed in a given amount of salt. The proposed flowsheet consists in first dissolving the fuel alloy in 63-37 mole %  $KF-ZrF_4$  until the composition 53.5-31.5-15.0 mole %  $KF-ZrF_4-AlF_3$  is attained, next adding  $KF$ , and then dissolving more fuel to attain the composition 54-20-26 mole %  $KF-ZrF_4-AlF_3$ . Laboratory tests of this process modification were successful.

On an engineering scale, sections of LITR prototype Al-U alloy elements were dissolved in 62-38 mole %  $KF-ZrF_4$  by using anhydrous hydrogen fluoride between 550 and 600°C in a 3.5-ID x 24-in. INOR-8 vessel. Two dissolutions (one partial, one complete) were made with no important difficulties. The second dissolution, in which a 2-in.-long, four plate, quarter cross-section LITR element was completely dissolved, was very smooth and rapid, as was the subsequent salt transfer. The penetration rates were 12 and 30 mils/hr for these dissolutions, respectively. The rates were calculated on the basis of the external surface only, since the internal channels between plates were closed by sludge at the end of the partial dissolution run.

Fluoride Volatility Processing: NaF Sorption Studies. -- The removal of  $UF_6$  and  $N_2$  by pelleted NaF was investigated by the use of differential-beds of pellets. Data on the rate and extent of sorption was obtained in the temperature range 29 to 100°C and in the  $UF_6$  concentration range 0.57 to 10.9 mole %.

The rate-controlling mechanisms were found to be transfer of the  $\text{UF}_6$  across a stagnant gas film surrounding the pellet, diffusion of the gaseous  $\text{UF}_6$  in the pore space of the pellet, and diffusion of the  $\text{UF}_6$  through a layer of  $\text{UF}_6\text{-NaF}$  complex covering unreacted  $\text{NaF}$  in the interior of the pellet. The crystalline density of the complex ( $\text{UF}_6 \cdot 2\text{NaF}$ ) was determined to be  $4.13 \text{ g/cm}^3$ , indicating that incomplete reaction of the  $\text{NaF}$  will occur for pellets in which the initial volume void fraction is less than 0.807.

A model was devised to represent the sorption of  $\text{UF}_6$  by a single pellet of  $\text{NaF}$ , and differential equations based on the model were written. A general method of solution of the partial differential equation describing simultaneous diffusion and irreversible reaction with variable diffusivity and reaction rate constant was derived for systems in which a steady-state type of solution is valid. The experimental data were correlated on the basis of the model, with a root-mean-square error of 9.5% for all points. The resulting computer code and associated data may be used for design of sorber systems such as fixed or moving beds.

The effective capacity of 1/8-in. right circular cylindrical pellets of  $\text{NaF}$  for  $\text{UF}_6$  was found to decrease with increasing temperature: At  $29^\circ\text{C}$  a capacity of 1.13 g  $\text{UF}_6$  per gram of  $\text{NaF}$  was observed, whereas the capacity at  $100^\circ\text{C}$  was only 0.64 g  $\text{UF}_6$  per gram of  $\text{NaF}$ . A model-predicted result of a 35% decrease in capacity for pellets having an initial void fraction of 0.45 was observed experimentally as the surface area was increased from 0.86 to  $1.1 \text{ m}^2/\text{g}$ .

In another study, a gasometric apparatus for rapidly characterizing dissociation pressures, sorption equilibria and kinetics, chemical states, and decomposition conditions was built and tested. The method simply involves the measurement of pressure changes, which indicate quantities of gases reacting with, or evolving from, known quantities of solid.

With the new apparatus, the dissociation pressures of  $\text{HF}$  and  $\text{UF}_6$  over their respective complexes with  $\text{NaF}$  and the decomposition of the  $\text{UF}_6$  complex to give  $\text{F}_2$  were verified. Additionally, a test for the sorption of  $\text{UF}_6$  on  $\text{NaF}$  was developed, the existence of the compound  $\text{Na}_2\text{UF}_8$  was

demonstrated, a more active form of NaF was made by a HF treatment, the stability of the pentavalent uranium-NaF complex was studied, data were obtained which indicated formation of a compound  $(\text{NaF})_2(\text{UF}_6)(\text{HF})$ , and the interaction of HF and  $\text{UF}_6$  on NaF was studied.

Fluoride Volatility Processing: Corrosion. -- Laboratory-scale studies were made at Battelle Memorial Institute to study the corrosion occurring during the cyclic hydrofluorination of Zr-U alloy and fluorination of the resulting  $\text{UF}_4$  in the same vessel, using the current zirconium flowsheet salt (initially 37.5-37.5-25 mole % NaF-LiF-ZrF<sub>4</sub>). This work supplemented engineering-scale studies previously reported (ORNL-3436, p 12).

Specimens of L-nickel, HyMu 80, INOR-8, Ni-5 Fe, and Ni-1 Al were exposed alternately to HF-salt and  $\text{F}_2$ -salt in an INOR-8 container. The study continued for ten exposure cycles, each consisting of 20 hr of HF treatment at 650°C, 2 hr of  $\text{F}_2$  treatment at 500°C, and 2 hr of helium treatment (1 hr during each temperature transition period).

According to micrometer measurements, Ni-5 Fe, INOR-8, and HyMu 80 were the most satisfactory, in that order; the maximum corrosion rates for all alloys (excluding L-nickel) were 5.2 to 9.0 mils/month. Container wall penetration rates were in the order of 6 mils/month---rather inconclusive because of the variation in initial wall thickness. No intergranular attack was noted in specimens of HyMu 80, Ni-5 Fe, and Ni-1 Al. L-nickel showed its usual intergranular attack, and INOR-8 coupons, as contrasted to the container, showed grain boundary modifications which were only revealed by etching. Bend tests caused separation of the L-nickel grains, but none for the INOR-8 specimens. Further details are reported in BMI-X-234.

Fluoride Volatility Pilot Plant. -- Outstanding overall decontamination factors were obtained during run R-8, one of a series made to demonstrate the recovery in the pilot plant of uranium from highly irradiated Zr-U alloy fuel elements. The six-month decay time for the fuel used in this run was the minimum anticipated for the Zr-U alloy processing demonstration. This program is scheduled for completion in calendar year 1963.

Overall (including dissolution and product recovery) decontamination factors (DF's) ranged from more than  $10^8$  to more than  $10^9$  for volatile Sb<sup>125</sup>, Te, Ru<sup>106</sup>, and from more than  $10^9$  to more than  $10^{10}$  for nonvolatile Zr<sup>95</sup>, Sr<sup>90</sup>, and Cs<sup>137</sup>. A DF of nearly  $10^{11}$  was obtained for Nb<sup>95</sup>, possibly the highest DF ever achieved for a single-cycle radiochemical process. In general, dissolution DF's were small compared with the recovery DF's. Dissolution DF's for volatile fission products were: Te, 7200; Sb<sup>125</sup>, 5; Ru<sup>106</sup>, 2800; and Nb<sup>95</sup>, 50. Strontium-90 and Cs<sup>137</sup> DF's were 2 and 1.5, respectively, and probably reflect the accuracy of the calculations rather than decontamination. Trace quantities of Ru<sup>106</sup> and Zr-Nb<sup>95</sup> were detected in the UF<sub>6</sub> product, but all other nuclides were below analytical limits of detection. The UF<sub>6</sub> product beta-gamma natural background is being evaluated to determine whether AEC specifications for returned UF<sub>6</sub> were met during the run.

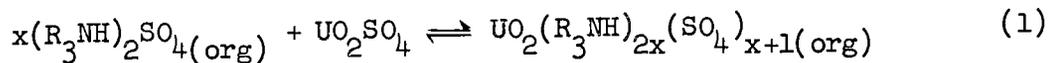
Total cationic impurities in the R-8 product were about 712 ppm (AEC total cation maximum is 300 ppm, based on uranium), with sodium, at 327 ppm, being the major impurity. Molybdenum removal by selective desorption from the movable-bed-absorber at 150°C was effective (the molybdenum level in the UF<sub>6</sub> product was 43 ppm, a fifth of the AEC maximum limit). The MgF<sub>2</sub> trap in the product recovery system removed Tc<sup>99</sup> and Np<sup>237</sup> to acceptable levels in the product (4 and 48 ppm, respectively).

#### CHEMISTRY RESEARCH

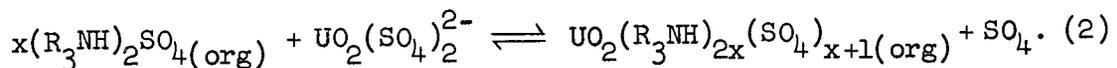
Separations Chemistry Research. -- Single-stage separation factors greater than 100 between the lanthanides cerium and europium and the transplutonium element americium were obtained by extraction with di(2-ethylhexyl)phosphoric acid (HDEHP) in hydrocarbon diluents from very dilute solutions of diethylenetriaminepentaacetic acid (H<sub>5</sub>DTPA) containing sodium nitrate. Europium was extracted much more slowly than were cerium and americium. Equally large separation factors between the two groups of elements, but without an observable time effect, were obtained in extractions by dialkylphosphoric acids and alkylhydrogenalkylphosphonates from mixtures of carboxylic acids and sodium salts of DTPA. Complete

separation of americium from cerium was accomplished by a reversed-phase chromatographic procedure in which a tracer-level mixture of the two elements in 1 M glycine nitrate, 0.1 M pentasodium diethylenetriamine-pentaacetate was filtered through a 20-cm-high column containing 5 ml of 0.3 M HDEHP in diisopropylbenzene absorbed in 5 g of microporous polyethylene. Residual americium was washed from the column with only about 2 column volumes of a similar unspiked aqueous solution.

From the inception of the Amex extraction of uranyl sulfate, there has been question as to whether the actual mechanism of extraction is transfer of a neutral complex,



or exchange of anions,



This question cannot be answered by any equilibrium measurement, since both mechanisms are thermodynamically equivalent. Kinetic measurements of the transfer of S<sup>35</sup>-tagged sulfate between amine sulfate organic solution and sodium sulfate aqueous solution, with uranium absent, present and being extracted, or already at equilibrium distribution, showed that both mechanisms contribute to the total uranium transfer. Mechanism (1) predominates in the extraction from low-sulfate solutions, where little of the uranyl ion is complexed (e.g., greater than 50% as uncomplexed UO<sub>2</sub><sup>2+</sup> at less than 0.01 M Na<sub>2</sub>SO<sub>4</sub>). Mechanism (2) becomes important at higher sulfate concentrations, where most of the uranium exists in anionic complexes (e.g., 75% as UO<sub>2</sub>(SO<sub>4</sub>)<sub>2</sub><sup>2-</sup> at 0.5 M Na<sub>2</sub>SO<sub>4</sub>).

Separations Process Development. -- In a continuous run in mixer-settler equipment, more than 98% of the cesium was recovered from an alkaline pollucite ore leach liquor (pH about 13) by extraction with 1 M 4-sec-butyl-2(α-methylbenzyl)phenol (BAMBP) in kerosene and stripping with hydrochloric acid. The strip solution contained 375 g of cesium per liter. Decontamination factors for cesium (from feed liquor to strip solution product) were 180 from Rb, 700 from K, more than 10,000 from Na, more than 700 from Li, and more than 10,000 from Si. The cesium was

also readily stripped from the extract with water and carbon dioxide in a slightly pressurized contacting vessel to give a cesium carbonate product.

Volatility Studies. -- Increasing the fluorine pressure used in tests on the volatilization of  $\text{PuF}_6$  from molten 31-24-45 mole %  $\text{LiF-NaF-ZrF}_4$  at  $600^\circ\text{C}$  resulted in a faster volatilization rate. In two tests at about 40 psig of fluorine pressure, half-time values of 1.3 and 1.7 hr were obtained. These are markedly less than the normal value of about 4 hr. This pressure effect indicated that the slow volatilization rate is basically due to unfavorable chemical kinetics.

No change in  $\text{PuF}_6$  volatilization rates were observed in other tests where 2 wt %  $\text{PbF}_2$  was added to the molten salt to act as a fluorine carrier, or where 50-50 mole %  $\text{LiF-ZrF}_4$  salt was used to avoid the possibly adverse effect due to the presence of  $\text{NaF}$  as a complexing agent.

#### PEACEFUL USES OF NUCLEAR EXPLOSIONS

Chemical Applications of Nuclear Explosions. -- Leaching of the water insoluble residue from a composite Gnome sample with hydrochloric acid gave almost the same solubility and percentage of activity leaching as was found in previous leaching experiments with nitric acid. With 30 ml of acid per gram, 80% of the plutonium and beta emitting rare earths were leached, and 75% of the solid dissolved at hydrofluoric acid concentrations of 1 to 6 N. By using only 6 ml of acid per gram, 60% of the plutonium and total rare earths were leached, and 50% of the original sample dissolved. Analyses for calcium and magnesium showed that nearly all the magnesium dissolved when 6 ml of acid were added for each gram of residue. However, the concentration of dissolved calcium showed a threefold increase when the acid-to-sample ratio was increased from 6 to 30 ml/g. Acid consumption was 15 meq/g. Glycolic acid was about 80% as effective as the equivalent concentration of hydrochloric or nitric acids for the leaching of plutonium and rare earths.

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