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CHEMICAL TECHNOLOGY DIVISION

MONTHLY REPORT FOR DECEMBER 1962

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ABSTRACT

Process Development studies are reported on Power Reactor Fuel Processing (Corrosion Studies, High Capacity Interim-23 Flowsheet, Protactinium Adsorption Studies, Hydrolysis of Uranium Carbides, Dissolution of PuO_2 Pellets, Shear-and-Leach Process Development); Thorium Utilization Program (Fuel Cycle Development: Fuel Preparation); Waste Disposal Development (High-Activity-Waste Calcination: Corrosion Studies, High-Activity-Waste Calcination: Fixation of Waste in Glass, Foam Separation Studies); Chemistry Research (Thorium Oxide Studies, Equipment Decontamination, Spectrophotometric Studies of Solutions at Elevated Temperatures and Pressures); and Isotope Development Program (Recovery of Fission Products).

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POWER REACTOR FUEL PROCESSING

Corrosion Studies. — Corrosion studies in support of the oxidation--ash-dissolution process for graphite fuels and of the chloride volatility process were continued. After three cycles in air at 800°C followed by treatment in boiling 5 M HNO_3, specimens of Corronel 230 showed either slight weight gains or weight losses corresponding to a corrosion rate of less than 0.1 mil/month. After one cycle, type 304L stainless steel specimens showed weight losses corresponding to corrosion rates of 0.3 to 1.6 mils/month. In flowing 1 M HF--0.06 M H_2O_2 at the boiling point, Corronel 230 showed overall corrosion rates of 13, 8, and 7 mils/month for specimens exposed to the vapor, at the interface, and to the solution, respectively; there was local pitting attack in welded areas.

In cyclic tests consisting of 5 hr in oxygen at 800°C followed by 5 hr in Cl_2-CCl_4 at 500°C, Nichrome V corroded at rates which increased from 5 or 6 mils/month for two cycles to 80 to 136 mils/month for 13 cycles. Exposure of Hastelloys B and C, "A" nickel, and Carpenter 20 to waste zirconyl chloride solution at room temperature resulted in corrosion rates of less than 0.1, less than 0.1, 0.4, and 0.6 mil/month during 264 hr of exposure. Types 304L and 309SCb alloys corroded at rates greater than 60 mils/month for 96 hr of exposure. At 50°C both Hastelloys continued to show rates of less than 0.1 mil/month for 96 hr exposure; rates for "A" nickel and Carpenter 20 were 10.5 and 9.0 mils/month, respectively.

High Capacity Interim-23 Flowsheet. — A laboratory experiment with 5% TBP as the extractant in an Interim-23 flowsheet (acid) resulted in decontamination factors of 4×10^4, 8×10^3, 3×10^4, and greater than

1×10^5 from gross gamma, ruthenium, zirconium-niobium, and total rare earths, respectively, and a uranium loss of 0.1%. An experiment with 10% tributyl phosphate (TBP) resulted in the formation of a third phase. The extraction capacity provided by an Interim-23 flowsheet, using 2.5% TBP, as is normal, resulted in approximately the same capacity for UO_2-ThO_2 fuel elements as that provided by the Acid Thorex process in a plant optimized for the Purex flowsheet. These experiments indicate that 5% may be the maximum TBP concentration that can be used successfully with the Interim-23 process and therefore provide the maximum capacity for thorium processing in a given set of equipment. This capacity is about twice that achieved with the Acid Thorex process. The capacity for the two systems would be the same in equipment optimized for the extraction process used.

Protactinium Adsorption Studies. — A series of 24-hr batch adsorptions on various amounts of 100- to 200-mesh unfired Vycor in contact with an 11 M HNO_3 solution of thorium (116 g/liter), uranium (5 g/liter), and protactinium (3.24×10^6 counts min^{-1} ml^{-1}) showed that the distribution coefficient of the protactinium was 3100; 5% of the protactinium was unadsorbable. Solutions containing 0.04 M F^- in addition to the other constituents, gave coefficients of about 200, with none of the protactinium unadsorbable. The addition of 0.1 M Al^{3+} to complex the fluoride increased the coefficient to 2900, with 6% of the protactinium unadsorbable. When zirconium (0.01 g/liter) niobium (about 0.01 g/liter) were added to the solutions, in addition to the fluoride and aluminum, the coefficient decreased to 1400 but the percentage of unadsorbable protactinium remained the same.

Hydrolysis of Uranium Carbides. — Basic chemical studies on the hydrolysis of uranium and thorium carbides in water and mineral acids

was continued. Although the products of the hydrolysis at 80°C of as-cast uranium carbides of compositions between UC and $UC_{1.86}$ showed a systematic decrease in the methane concentration and an increase in the amounts of higher hydrocarbons, free hydrogen, and wax produced as a result of increasing carbon concentration in the alloy, the amount of methane evolved was considerably less than expected from the calculated amount of UC present in a UC-$UC_{1.86}$ or a UC-$UC_{2.00}$ binary mixture. Since both x-ray and metallographic evidence indicated that the as-cast specimens having compositions between UC and $UC_{1.86}$ were binary mixtures of the mono- and dicarbide, it would appear that the carbon units in the monocarbide crystal react preferentially with hydrogen to produce methane during hydrolysis, but if dicarbide is also present in the solid, it reacts with other carbon units to produce higher hydrocarbons, and with hydrogen to produce methane.

Dissolution of PuO_2 Pellets. — The instantaneous dissolution rate of PuO_2 pellets (90% of theoretical density) in boiling mixtures of HNO_3-HF-$Al(NO_3)_3$ at an Al/F mole ratio of 3 is proportional to the 0.45 power of the hydrofluoric acid concentration.

Shear-and-Leach Process Development. — The UO_2-ThO_2 particles smaller than 9520 μ dislodged from 0.5- and 1.0-in. sheared lengths was 90.7 and 39.1 wt %, respectively, of the original core material in the assembly, compared with 84.5 and 35.4 wt % for UO_2. Particle-size measurements from 0.5- and 1.0-in. sheared lengths of stainless-steel-clad unirradiated ThO_2-UO_2 ORNL Mark I fuel assembly show that there is an average of 3.5 and 2.6 wt %, respectively, more core material found in the particle-size range less than 9520 μ to less than 44 μ than was

observed on the shearing of UO_2-filled assemblies. Similarly the amount of stainless steel cladding reduced to less than 1000 μ in size was 0.23 and 0.05 wt %, and 0.38 and 0.10% for ThO_2-UO_2 and UO_2, respectively.

The conveyor-feeder and leacher were decontaminated (from natural uranium) and dismantled preparatory to the installation of larger-diameter feed chutes on each unit, and a steam jacket on the inner drum of the leacher. The ram of the shear was removed and machined to larger tolerances to allow better adjustment of clearance between the fixed and moving blade with the wedge-shaped gibs. Inspection of shear components following 13 chop-leach runs with stainless-steel-clad UO_2 assemblies showed no evidence of wear on the stepped blade, gibs, liners, or connecting rod. The shear had made about 1600 cuts on UO_2-filled Mark I prototype fuel assemblies containing 36 tubes per assembly during the 13-run campaign.

THORIUM UTILIZATION PROGRAM

Fuel Cycle Development: Fuel Preparation. — Spheroidal thorium oxide and 3 at. % uranium--thorium oxide particles, 100 to 200 μ in diameter, were prepared by dispersion of the oxide aquasol in carbon tetrachloride, then "setting" the particles with isopropyl alcohol and drying to gel. The particles retained their rounded-gel shapes when fired to 1150°C in air.

Steam denitration of uranyl nitrate was investigated as a method for the preparation of active UO_3 hydrate. A carrier gas (air, bubbled through water at controlled temperatures) was passed over the uranyl nitrate in a rotary calciner, giving various hydrated UO_3 products at temperatures from 175 to 300°C in about 5 hr. Products had crystallite sizes of 166 to 850 A. The most active oxide was the β-$UO_3 \cdot H_2O$ prepared at 175°C.

In six successive batches (a total of about 35 kg) of 3 mole % UO_2--ThO_2 prepared by the Kilorod sol-gel procedure, the uranium was distributed within the specified 3 ± 0.03 mole % for all particle-size fractions. The addition of 4-1/2% U as UO_3 or $(NH_4)_2U_2O_7$ to a mixed urania-thoria sol gave a 7-1/2% UO_2--ThO_2 sol-gel product without excessive uranium segregation. Calcination in Ar—4% H_2 gave products having O/U ratios of 2.019 to 2.026, and in pure nitrogen or argon, 2.099 to 2.31. Residual gases released at 1200°C in vacuum were 0.004 to 0.018 cc/g for the coarse fractions and 0.067-0.130 cc/g for the fines after calcination. Grinding and sizing increased the amount of evolved gases to 0.012 to 0.021 cc/g for the coarse and 0.170-0.320 cc/g for the fine fractions.

A product prepared with 6% enriched uranium for irradiation specimens was analyzed for 24 trace elements after crushing and sizing. The macroscopic cross section of total impurities was equivalent to 5 ppm of boron, with iron, sodium, and nickel as the principal impurities. Two 22.5-in.-long irradiation capsules were sent to the Materials Testing Reactor for irradiation of the 5 wt % enriched uranium material (sol-gel UO_2-ThO_2) to 120,000 Mwd per metric ton of thorium.

WASTE DISPOSAL DEVELOPMENT

High-Activity-Waste Calcination: Corrosion Studies. — Specimens of types 304L and 347 stainless steels corroded at rates of less than 0.1 mil/month in the superheated vapor above refluxing simulated Purex waste solution, compared with rates of 2.6 and 3.3 mils/month, respectively, for specimens in the condensing vapor. Type 304L stainless steel underwent elongation of between 9.2×10^{-5} and 5.4×10^{-5} in./in. while subjected to a Purex evaporation-fixation cycle followed by about 165 hr at 1050°C. Welded specimens of various stainless steels exposed for 31 days at about 575°C in the Carey salt mine, Hutchinson, Kansas, corroded at rates of 0.45 to 1.3 mils/month.

High-Activity-Waste Calcination: Fixation of Waste in Glass. — Melts containing 39 to 43% waste oxides from simulated high-sulfate Purex waste retained about 95% of the original sulfate when heated to 100°C above the softening points for 100 min. Ruthenium volatilized to the extent of about 24% from semiengineering-scale fixation-in-glass experiments with simulated TBP-25 wastes. Also, about 77% of the mercury volatilized.

Foam Separation Studies. — The effects of column diameter, sludge height, and flow rates on the precipitation of calcium and magnesium from ORNL low-activity waste were studied. Column diameters varied from 2 to 9 in., and flow rates from 7.4 to 60 gal ft^{-2} hr^{-1}. Within this range the hardness of the waste water was reduced from about 100 to 2 to 4 ppm (as $CaCO_3$) when precipitation was induced by making the water 0.005 M each in NaOH and Na_2CO_3. An apparatus was tested for the removal by foaming of the surface active agent sodium dodecylbenzene sulfonate; 90% was separated from a solution containing the above-listed chemicals plus 50 ppm of the surfactant.

Engineering studies were continued in 6-in.-ID columns, using dodecylbenzenesulfonate (DBS) as the surfactant. One liquid-feed distributor gave HTU_x values of less than 2 cm for flows of 120 gal ft^{-2} hr^{-1} or less; the HTU_x values were 2 to 20 cm for other distributors or higher flow rates. The difference in HTU_x values for the spinnerette gas spargers with 50- and 80-μ-diam holes was small, variable, and probably not significant. Recovery of DBS by simple foaming could be applied to give effluents of about 5 ppm of surfactant for 0.02 N Na^+, and about 20 ppm for 0.001 N Na^+. Process water was used to simulate low-activity waste. The calcium and magnesium were precipitated, and the slurries were used as feed for the foam separation column. Decontamination factors of 81 to 162 for strontium obtained with $CaCO_3$-$Mg(OH)_2$ slurry feed were reduced to less than 3 when 10 ppm of FAB household detergent was added to simulate the effect of such detergents in low-activity wastes.

CHEMISTRY RESEARCH

Thorium Oxide Studies. — Examination of the thorium oxide preparations irradiated in the multiple-sample, 12-month LITR irradiation experiment is continuing (see October Monthly Status and Progress Report). Special attention was paid to the code P-82 thoria pellets. Those pellets, when irradiated dry, lost less than 0.05% of their weight; those irradiated under D_2O lost less than 0.4%; and those irradiated in the D_2O vapor lost 6.2%.

Wear rates in spouting-bed tests of the dry- and D_2O-irradiated pellets were only about two-thirds those of the original or D_2O-control pellets throughout 6-hr tests. Pictures of the irradiated pellets after the wear tests showed that, with the exception of an occasional pit, the wear rate was uniform and indicated little radiation-induced structural damage.

Impact fracture tests were carried out on the cold and the irradiated P-82 pellets. The fracture test consisted in dropping a pestle (in a pattern of increasing heights) down a loose-fitting tube onto a pellet in a mortar. The original pellets, those from the 12-month D_2O-control experiment, and those from the 3- and 12-month irradiations (dry and in D_2O) showed impact fracture energies between 0.651 and 0.945 in.-lb. The pellets that had been irradiated for 12 months in D_2O vapor had a fracture energy of 0.342 in.-lb. There was further evidence of the structural weakening that resulted from the vapor-phase irradiation: Unlike the response of pellets irradiated under D_2O, stirring of the vapor-phase-irradiated pellets in water resulted in some breakage.

The interiors of the 12-month liquid-phase-irradiated pellets were similar in color to those of the unirradiated pellets, while those of the dry-irradiated pellets were black. Heating at 400°C for 1 hr in air failed to change the color, but heating at 900°C overnight restored the original color. No weight increase was noted as a result of the 900°C heating, and it is postulated that the blackening resulted from a very slight oxygen deficiency.

Definitive pore-size-distribution data (according to the nitrogen-adsorption method) for pellets that had been irradiated for three months in D_2O and for some irradiated for an extended time in 260°C water indicated that irradiation in D_2O decreased the nitrogen-measured void volume from a value of 0.6 to 0.4%. It produced a large number of small pores (of about 30-A radius), probably associated with individual fission events, and removed larger pores of about 100- to 400-A radius. Since both the irradiated and unirradiated pellets had densities less than 97% of theoretical, most of the surface-connected void volume must be associated with pores of greater than 1100-A radius.

Equipment Decontamination. — The steam-jet decontamination of stainless and carbon steels that had been baked in helium at 500°C, simulating severe exposures in gas-cooled reactors, was very satisfactory when oxalate-peroxide reagents were aspirated into the jet at a proportion of about 16% of the steam weight. In 30 min, carbon-steel decontamination factors for strips that were 2 in. from the jet were 1.2×10^4 for $Ba\text{-}La^{140}$, 5.4×10^3 for $Ru\text{-}Rh^{106}$, and 635 for $Zr\text{-}Nb^{95}$. At 24 in. from the jet the factors were 39, 285, and 180, respectively.

The corrosion rates on carbon steel were only 0.006 and 0.002 mil/hr in the two positions so long as sufficient hydrogen peroxide was used to maintain metal passivity (0.5 M or over in the feed), but the rates increased sharply at lower peroxide concentrations. The decontamination of stainless steel was less effective than that of carbon steel, but after 2 hr in the steam jet with several oxalate-peroxide reagents of low corrosivity the $Zr\text{-}Nb^{95}$ decontamination factor averaged 90 on eight stainless steel coupons spaced 2 to 24 in. from the jet nozzle.

One type of cold-rolled carbon steel was brilliantly polished and decontaminated by a factor of 170 from 500°C-baked $Ce\text{-}Pr^{144}$ by a 5-min immersion in 0.5 M oxalate--0.01 M F^---0.53 M H_2O_2 at pH 3.0, at 60 to 70°C, with a removal of 1.5 mils of surface. The steel had previously been treated with other decontaminating solutions. The polished surface was rust-resistant for several days in water at room temperature. The solution did not polish case-hardened steel, however. Sulfate solutions showed little promise as decontamination solutions for carbon steel because of their high corrosivity.

Spectrophotometric Studies of Solutions at Elevated Temperatures and Pressures. — As an important adjunct to this program, a method was devised for measuring the densities of aqueous solutions at accurately measured temperatures and pressures up to the solution critical points. A knowledge of solution densities is mandatory for the calculation of the ionic molar concentrations in solution at temperatures from 25°C up to the solution critical point (about 372°C). The liquid volume of a weighed solution of known composition is determined in an autoclave of suitable material, and an x-ray photograph is made to show the position

of the vapor-liquid interface in a calibrated section of the autoclave. The density at any temperature is determined from the weight of solution and the location of the interface at that temperature.

A prototype high-pressure autoclave was designed and then built entirely of pure titanium. It can be operated up to 4000 psi at 400°C, and it consists of a bulb section of about 4-ml volume containing an internal thermocouple well, a uniform expansion section about 12 in. long, and a high-pressure closure head with a fitting that contains a titanium capillary that leads to a pressure transducer. Markers on the expansion section facilitate the measurement of the solution interface location by x-ray measurements. The densities may be determined at 370 to 371°C, within a maximum error of $\pm$ 0.6%, assuming the worst error interaction. (At somewhat lower temperatures the error will be less.)

A special furnace and the necessary control instrumentation were designed and constructed to contain the autoclave in an essentially isothermal state over its entire length of about 18 in., up to temperatures to at least 375°C. The furnace is also slotted so that unobstructed x-ray photographs can easily be obtained. A motor driven x-ray film cassette and support system has also been designed and constructed. The x-ray film can be driven past the slit in the system behind the autoclave from a rate of about 0.05 in./min, up to about 3 in./min. Two modes of operation are being investigated: operation at a series of isothermal temperature plateaus, or by a continuous change of film position and autoclave temperature simultaneously, to yield, in effect, a plot of interface height and autoclave temperature. All the equipment is being installed and will be used with a 300-kv Norelco x-ray system.

Computer techniques are being utilized to make possible the analysis of absorption spectrophotometric data from many different types of systems and reactions. The systems can contain a number of different components; each component can be represented by several absorbing species. The species that may or may not be at equilibrium can be exactly analyzed. In the nonequilibrium case, the concentrations of the species can change significantly during the time interval that is required to obtain an individual spectrum, and conventional analysis techniques are not applicable to the multipomponent, nonequilibrium systems. From a series of spectra obtained for a given system at various times, an iterative technique is used to obtain the reaction-rate curve for each absorbing species present.

The detailed problem description for a computer program of very general applicability has been prepared and is being programed by the Mathematics Division. A summary report on this subject will be prepared. From the necessary input data the program sets up the equations which are required to calculate the concentrations for all of the absorbing species at any given time. In order to use the program, it is necessary to obtain absorption spectrophotometric measurements for at least as many different wavelengths as there are absorbing species in the system under study; a greater number of wavelengths can be used. The molar absorptivity for each species must be known at each wavelength at which a measurement is made. For systems not at equilibrium, the times after reaction initiation at which the absorption measurements are made must also be known. The computed reaction rate data and other types of data are tabulated for each species if desired, and the rate curves can be plotted on the CALCOMP plotter as direct output from the computer, on either a concentration or mole fraction basis.

The program can handle almost all types of kinetic data, e.g., oxidation-reduction, disproportionation, combination, decomposition, and valence-state distributions, etc. A series of data from a given system that is unrelated kinetically, or kinetically slow reactions can also be analyzed. Within the program many options are available, including the use of least-squares techniques both before, during, and after convergence, and tabulation-plotting out-put options. Converged data may also be punched out to be used as input for more detailed kinetic analysis by subsequent programs.

Pa Chemistry. — Further studies of Pa distribution between sulfuric and acid solutions and Dowex 1 have verified that apparent distribution coefficients are dependent on the amount of Pa present. As aqueous Pa concentrations were increased above a critical value nearly all the added Pa went into the Dowex 1 resin phase. However, as the loading of Pa in the resin increased the Pa concentration in the aqueous in equilibrium also increased, but relatively very much more slowly. Stated differently, above a critical Pa concentration the apparent distribution coefficient increases very rapidly as more Pa is added. The critical concentration in the aqueous phase increases with the square of the sulfuric acid concentration, from 2.3×10^4 αc/m/ml in 1.5 N sulfuric acid to 1.1×10^6 αc/m/ml in 10 N acid.

ISOTOPE DEVELOPMENT PROGRAM

Recovery of Fission Products. — Continued studies of substituted phenols as extractants for the recovery of cesium from waste solutions showed that a commercial phenol, 2-benzyl-4-chlorophenol (Monsanto Santophen-1), has the highest cesium extraction power of any phenol tested so far. In a batch countercurrent test with 1 M Santophen-1 in diisopropylbenzene, 99.8% of the cesium was recovered from simulated Purex 1WW solution (tartrate-complexed and adjusted to pH 12.3) in 6 extraction and 2 scrub stages (the scrub solution was 0.01 M NaOH, 0.002 M sodium tartrate) at feed/organic/scrub ratios of 6/3/1. The cesium was quantitatively stripped in a single contact with a 0.05 volume of 0.05 M HNO_3, resulting in an overall cesium concentration of 40 and an overall cesium-from-sodium separation factor of about 6×10^4. In a similar test with 0.6 M Santophen-1, more than 97% of the cesium was recovered at feed/organic/scrub ratios of 5/5/1. As in the previous test, the cesium was quantitatively stripped, resulting in an overall cesium concentration of about 20 and overall separation factors of about 6×10^4 from sodium and about 260 from rubidium.

Further tests with 4-sec-butyl-2(α-methylbenzyl)phenol (BAMBP) show that it has excellent selectivity for cesium over sodium, other fission products, and corrosion products, and has an almost negligible loss (≤0.4 g/liter) to adjusted 1WW solution.

Preliminary tests showed certain branched-chain carboxylic acids, for example, neo-tridecanoic acid, extract strontium preferentially to sodium from basic sodium nitrate solution. The strontium extraction increases rapidly above pH 7, reaches a sharp maximum between pH 8.5 to 9.5 and then decreases at higher pH's.

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