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ABSTRACT

Process development studies are reported on Fuel Cycle Development and Fuel Preparation, Power Reactor Fuel Processing, Radioactive Waste Treatment and Disposal, Equipment Decontamination, Protactinium Chemistry, Thorium Oxide Studies, Transuranium Elements Studies, U-232 Preparation, and Fission Product Recovery.

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FUEL CYCLE DEVELOPMENT AND FUEL PREPARATION

A 1.4-kg batch of 3 wt % uranium-thorium oxide was prepared by the sol-gel process, in which uranium was added as ammonium diuranate for compaction experiments by Babcock and Wilcox. The preparation was easily sized in the dried gel stage to fractions pre-estimated to give the size distribution desired when fired. Particle densities averaged 9.88 g/cc; the O/U ratio was 2.032 to 2.040; the gas release at 1200°C in vacuum was 0.003 cc/g. A test compaction in a 5/16- x 11-in. tube gave a packed density of 8.6 g/cc.

Further laboratory-scale experiments demonstrated the ability of the sol-gel process to produce particles with densities >9.8 g/cc, which can be compacted to bulk densities >8.7 g/cc when uranium is added as uranyl nitrate solution. Adjustment of the pH of mixed uranium-thorium oxide sols to approximately 3 either by ammonia addition or by controlling total nitrate and thorough mixing are required to prevent inhomogeneity with respect to U/Th ratio. Scale-up (15 kg) demonstrations have not produced satisfactory products reproducibly as yet.

The optical density of thoria sols at 6700 A wavelength varies linearly with thoria concentration, and for a particular thoria preparation shows a reproducible minimum with varying NO_3^-/ThO_2 mole ratio. This minimum is related to the surface area and to the maximum dispersion of the ThO_2 preparation.

In 15-kg-scale steam denitration test runs in the rotary calciner, ThO_2 products were produced which gave excellent thoria sols and uranium-thorium oxide products.

POWER REACTOR FUEL PROCESSING

Corrosion Studies. Unwelded Corronel 230 exposed to boiling 20 M HNO_3 was corroded at maximum rates of 5.5, 1.5, and 1.2 mils/mo in 720 hr in vapor, interface, and solution positions, respectively. All specimens underwent intergranular attack. In boiling 10 M HNO_3—0.5 M HF (116°C) in 72 hr, uniform rates of 42.6, 43.7, and 62.0 mils/mo for vapor, interface, and solution specimens, respectively were noted; at 60°C corresponding rates were 0.92, 1.73, and 2.37 mils/mo. In boiling 10 M HNO_3—0.5 M

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NaF—0.75 M $Al(NO_3)_3$, 408-hr maximum rates were 0.78, 0.71, and 0.71 mil/mo. Corresponding rates for welded titanium-45A were 0.02, 0.16, and 0.28 mil/mo. Neither alloy showed localized attack. In boiling 1 M HF, 48 hr exposure of Corronel 230 showed rates of 4.09, 23.2, and 43.7 mils/mo in vapor, interface, and solution, respectively. In 120 hr exposure to boiling, flowing 1 M HF—0.06 M H_2O_2, corresponding rates were 12.7, 10.6, and 2.13 mils/mo. Corronel 230 dissolved at a nominal rate of 2700 mils/mo in 4 hr exposure to boiling (104°C) 1 M HNO_3—3 M HF, while type 309 SNb was corroded at a maximum rate of 590 mils/mo. At 60°C, 72 hr exposure of Corronel 230 resulted in rates of 10.6, 0.67, and 1.25 mils/mo, respectively.

Corrosion rates of Inor-8 in chlorine gas at 508 and 720°C were 0.62 and 103 mils/mo, respectively, in 24 hr. These rates are lower by a factor of about 10 than those previously found for Haynes 25 and Nichrome V.

Titanium-45A exposed 120 hr to a boiling solution prepared by dissolving stainless steel to 60 g/liter in 2 M HCl—5 M HNO_3—0.1 M HBF_4 was covered with a hard, adherent deposit. After descaling, corrosion rates were 0.26, 6.02, and 12.5 mils/mo for vapor, interface, and solution specimens, respectively.

Welded specimens of commercial Ni-o-nel and LCNA exposed in vapor and condensate above boiling 4 M HNO_3 for 192 hr were corroded at rates of 0.15 and 0.01 mils/mo, respectively, for Ni-o-nel and at 0.06 and 0.01 mil/mo for LCNA. Both alloys in the vapor phase assumed a brown stain.

Darex Process. In the Darex dissolution of APPR fuel, which contains ~2% silicon, voluminous quantities of insoluble silica are formed which become highly hydrated during chloride removal and plug the chloride stripping column. The silica in a solution containing 60 g of stainless steel per liter made by dissolving APPR fuel in 5 M HNO_3—2 M HCl containing 0.005 M HBF_4 was made filterable by digesting 2 hr at 90°C. At a filtration rate of 0.5 ml/cm^2·min, silica was 99% removed. Digestion without filtration led to 87% passing through the chloride stripper column, the remainder being easily removed from the column by washing with dilute nitric acid. In both cases, ~17% of the fluoride was volatilized during chloride stripping. Boiling the dissolver solution for 24 hr at total reflux also converted the silica to a form that was 99% removed by filtration at an average rate of 1.2 ml/cm^2·min. Insoluble stainless steel

carbides and oxides were also removed quantitatively. Addition of gelatin or molybdate to the dissolver solution formed sticky, gelatinous precipitates.

Graphite and Uranium Carbide Fuel. Digestion of type GBF graphite with boiling 90% HNO_3 for 50-100 hr oxidized <2% of the graphite. A small amount of dark red-brown, soluble organic oxidation products was isolated by evaporating off the nitric acid. These products were acidic, with neutralization equivalents of about 88 g/eq, indicating that their molecular weights were much higher than those of the products isolated after extended oxidation.

Small castings of uranium monocarbide were analyzed for uranium, tungsten, total carbon, free carbon, and oxygen, subjected to the standard hydrolysis tests, and examined metallographically. The high purity of the castings, estimated as 89% UC, 5% U_2C_3, 4.5% U metal, 1.3% UC_2, and 0.2-0.4% WC, renders them suitable for irradiation and use in studies of the effect of radiation on the chemistry of UC.

Mechanical. In further shear-leach tests, a mockup fuel assembly of 36 porcelain-filled stainless steel tubes was easily sheared into 1/4-, 1/2-, and 1-in.-long pieces with the 250-ton prototype shear using a stepped blade. Discrete pieces of tubing were produced in the 1/2- and 1-in.-long cuts, but the 1/4-in. pieces were severely flattened, entrapping core material.

Solvent Extraction. Laboratory studies with synthetic U-233 solutions indicated that a countercurrent extraction and static scrub system would satisfactorily decontaminate uranium from Th-228 for use in fuel recycle studies. Uranium extracted from 1 M $Th(NO_3)_4$—0.8 M $Al(NO_3)_3$ with 2.5% TBP in Amsco or 2.5% di-sec-butyl phenylphosphate in diethylbenzene and scrubbed with 4 M NH_4NO_3 was decontaminated from Th-228 by factors of 700 and 5×10^4, respectively. The decontamination with DSBPP without scrub was 600. When the feed contained only 0.25 g/liter thorium, decontamination factors could not be determined due to the uncertainty of thorium determinations in very dilute solutions.

A series of irradiated ThO_2 washing tests with 0.5 to 4 M HNO_3 failed to remove a substantial amount of activity from the ThO_2, 0.2% at room temperature and 0.7% at 95°C.

Zirflex Process. Sixteen Zircaloy-2-clad UO_2 pellets, irradiated in the NRX to about 20,000 MWd/t and decayed about nine months, were declad with boiling 6 M NH_4F—1 M NH_4NO_3. The pellets were fractured but did not fall apart. Uranium losses were 0.03-0.08%; the plutonium losses were 0.01-0.04%. The core pellets were 99.5% dissolved in 4 M HNO_3—0.1 M $Al(NO_3)_3$ in 5 hr, which was slightly faster than the rate for unirradiated pellets.

RADIOACTIVE WASTE TREATMENT AND DISPOSAL

Engineering, Economic, and Hazards Evaluation. Space requirements to dissipate the fission product heat in the waste from a 56,000-Mw_t nuclear power economy were calculated for cylinders of calcined waste buried vertically in the floors of rooms in salt media. Net area requirements calculated for Purex wastes in acidic and reacidified forms in 6-, 12-, and 24-in.-dia vessels range from 20 acres/year after 0.65 year to about 2 acre /year at 30 years age at burial. Corresponding results for Thorex wastes are 10 and 0.7 acres per year. Calculations were made also for an acidic Thorex glass in 6-in.-dia vessels. The primary effects of the form of the waste (acidified, reacidified, glass) and vessel diameter are to limit the minimum burial age, although area requirements may be affected by factors of 2-3 by waste form and vessel size near the minimum burial ages.

High-Level Waste Calcination. The product of semicontinuous evaporation and calcination of simulated TBP-25 waste to form a lead phosphate glass (additives: 2 moles NaH_2PO_2 and 0.25 mole PbO per liter) in a 24- x 4-in.-dia stainless steel pot had a density of 2.84 g/ml and a volume about 1/8 that of the concentrated (1.8 M Al) waste solution. The thermal conductivity of the glass varied from 1.05 Btu/hr·ft·°F at 300°F to 1.60 Btu/hr·ft·°F at 1050°F; the temperature coefficient, which was positive, was nonlinear with temperature, indicating an amorphous solid. Ruthenium volatility decreased from 13.7 to 3% during processing, indicating removal of ruthenium contamination of the equipment from earlier experiments.

Stainless steel, type 304, should be a satisfactory material for waste calcination pots, with negligible corrosion in storage. Corrosion was maximum during the period when the last of the acid and water were being driven off at 850-1000°C. Overall corrosion in 24 hr evaporation-calcination of Purex and TBP-25 wastes was 0.26 to 0.34 mil at 900°C (8-10 mils/mo). Of this about 0.4 mil was attributable to air oxidation. When glass-making additives were present, rates were about 42 mils/mo for the TBP-25 lead phosphate glass at 920°C and 145 mils/mo for a Purex borophosphate glass at 900°C. Release of SO_3 from the Purex caused aggressive intergranular attack (possibly catastrophic) at temperatures above 900°C, the critical temperature, depending on the melt composition.

In 11 tests with simulated Darex, TBP-25, and TBP-25 wastes containing organic residues, average overall evaporation-calcination rates for the full-scale (96 x 8-in.-dia) pot calciner decreased with increased filling (evaporation) time. This is attributed to the higher bulk densities and consequently higher thermal conductivities in the final product. Both batch and continuous flowsheets were demonstrated along with satisfactory control of the continuous evaporator. Filling rates averaged 8.6 to 19.4 liters/hr, depending on feed type, concentration, and filling period, with final solid product bulk densities ranging from 0.61 to 1.42 g/cm^3.

Material and energy balance calculations for a hypothetical Purex waste indicated that an 8-in.-dia pot must be used for wastes cooled less than 180 days, but that a 12-in.-dia pot may be used for calcining older wastes. The maximum vapor rates to the fractionator for Purex wastes are ~20% greater than those predicted for TBP-25 but the column will operate below flooding at the higher rate.

Low-Level Waste Treatment. Laboratory-scale support studies for the low level waste (essentially tap water) treatment pilot plant showed that (1) BO-4 grade vermiculite is subject to plugging and complete stoppage if allowed to contact small amounts of solids; (2) cobalt and ruthenium removal from the waste stream is improved by the addition of an anion exchange column to the system; (3) Nalco reagents are satisfactory, 1 ppm accuracy, for pilot plant control analysis; and (4) residual hardness of the liquid leaving the clarifier should be decreased by densification of the sludge blanket by recirculation.

Four demonstration runs on ORNL waste were completed in the low-level waste treatment pilot plant. In all runs, Sr-90 and Cs-137 were removed from the solutions by decontamination factors of >2000 and >246, respectively. The plant effluent contained <1.5% of the current MPC_w values for these isotopes for a 168-hr week. Run times were from 71 to 100 hr of continuous operation, with the use of 1800-2100 resin bed volumes.

EQUIPMENT DECONTAMINATION

An electrode potential method was developed for determining the concentration of hydrogen peroxide necessary to induce metal passivity at all formulations and temperatures. Oxalate-peroxide decontaminating solutions were found which would be slightly corrosive at room temperature when rubbed on mild steel with a rag or sponge, but to which the steel would become passive when rubbing ceased. A typical solution of this type, which also dissolved ordinary rust, was 0.4 M ammonium oxalate—0.2 M ammonium acetate—0.7 M H_2O_2 at pH 2.0.

Acetate is unsuitable as a buffer in oxalate-peroxide solutions for use on carbon steel at elevated temperatures because of the corrosive nature of the acetic acid in the vapor above the solution. Nonvolatile buffers such as citrate and glycolate may be used instead, or suitable acid reserve may be obtained by lowering the pH. In the latter case, corrosion may then be controlled by lowering the temperature and raising the peroxide concentration. For example, the corrosion rate in 0.4 M oxalate—0.4 M H_2O_2 at pH 3.0 and 75°C was 6 mils/hr, but when the peroxide concentration was raised to 1.5 M, dropped to 0.001 mil/hr. Formulas with a higher pH, with the acid reserve provided by a nonvolatile buffer, offer greater safety against corrosion when peroxide concentration drops than those at lower pH with higher peroxide concentrations.

PROTACTINIUM CHEMISTRY

The separation of uranium from protactinium in HCl-HF solutions by amine extraction was shown to be practical in laboratory tests. In 8 N HCl the distribution coefficient (O/A) for protactinium extracted into

0.1 N trilauryl amine in diethyl benzene decreased with increasing HF concentration from 0.9 in 0.01 N HF to 7×10^{-4} in 4 N HF. The corresponding uranium distribution coefficients showed a more complex dependence on HF concentration, decreasing from 300 in 0.01 N HF to about 45 in 0.26 N HF, then increasing to about 100 in 0.8 N HF, and finally decreasing with further rise in HF concentration. Separation of uranium and protactinium with 0.1 N trilauryl amine was best from 8 N HCl—0.8 N HF. The separation factor was 8×10^4 with one equal-volume extraction of a solution containing initially 9 mg/ml Pa and 2 mg/ml U.

THORIUM OXIDE STUDIES

The catalytic activity for H_2 and O_2 combination was determined for a slurry prepared from some of the dried, irradiated solids recovered from loop L-2-27S. The specific activity under a large excess of O_2 at 280°C based on palladium concentration was at least as high as and possibly higher than those obtained with other slurry-palladium catalyst systems which had not been irradiated and more than sufficient to account for the low radiolytic gas pressures observed during in-pile operation of the loop. At H_2/O_2 ratios ≥ 0.7 and after H_2 pretreatment, specific activities were much higher, in accord with previous experience with unirradiated palladium catalyst-slurry systems. Hence, the simultaneous reactor irradiation and pumping under O_2 apparently did not result in any deleterious effect on catalytic activity.

Surface area and porosity data were obtained on the code P-82 thoria pellets irradiated 3 months in D_2O at 250°C in the LITR ($\sim 3 \times 10^{13}$ n/cm^2·sec) and on pellets heated in water at 260°C for approximately 2000 hr without irradiation. The irradiated material had a surface area (BET) of 0.32 m^2/g, a void volume of 1.4%, an average pore radius of 219 A and a median pore radius of 70 A. The unirradiated pellets had a surface area of 0.023 m^2/g, a void volume of 0.6%, an average pore radius of 758 A, and a median pore radius of 344 A. Fifty-nine percent of the void volume of the irradiated material was in pores of radius $\leq$ 98 A and only 15% of the void volume of the unirradiated material was in pores of radius $\leq$ 112 A. If the data are correct, irradiation of the pellets under water apparently

greatly increased the specific surface area, somewhat increased the void volume, and produced a large number of small pores. Despite the increase in surface area and void volume, wear resistance of the irradiated pellets (1 hr standard spouting bed test) was about a factor of 10 better than that of the unirradiated pellets (see October 1961 Monthly Progress Report).

TRANSURANIUM ELEMENTS STUDIES

Investigation of the solvent extraction chemistry of 2-ethylhexylphenylphosphonic acid (2-EH(ØP)A) was continued. Both UO_2^{2+} and Pu^{4+} were strongly extracted from HCl of all concentrations. Their distribution coefficients are 10^2 to 10^3 greater than that of Cf^{3+}. Distribution coefficients between 1 M 2-EH(ØP)A in diethyl benzene and HCl for cerium, europium, terbium, and thulium were minimum at 5.5, 6.5, 7.5, and 8 N HCl, respectively. Separation factors between adjacent lanthanides remained constant to the minima and began then to converge at higher acidities.

Californium was separated from americium in a countercurrent batch extraction run for a californium recovery of >99.5% with an americium removal of >99.9%. The conditions were: 3 scrub and 4 extraction stages; feed, 1.4 N HCl; scrub, 1.8 N HCl; extractant, 1 M 2-EH(ØP)A in diethyl benzene; and feed to scrub to extraction volume ratios of 1:1:2.

Results from a disconnect selection test program indicated that either of two designs is about equally acceptable. Leak-tight closures were obtained for repeated assemblies, for interchanged halves, and for misalignment and seal surface marring intended to exceed that expected for normal use. In accelerated corrosion tests with aqua regia, the connecting lines failed before the seal surfaces leaked.

Use of a modified impact wrench and manipulators was demonstrated for remote disassembly and/or removal of a gang disconnect, an air-operated valve, and a diaphragm pump. A water sprinkler satisfactorily extinguished an o-xylene fire in a glove box even when the front was removed to increase the intensity of the fire. A 0.82-in.-i.d. pulsed column with 30% Alamine 336 in diethyl benzene with 11 M LiCl showed stage heights of 16.5-19 in.

for scrubbing europium from the solvent and flooding rates of 9 ml/min organic—3 ml/min aqueous at 25°C and 18 ml/min organic—6 ml/min aqueous at 50°C.

U-232 PREPARATION

Approximately 6.8 mg of U-232 was prepared and partially purified. Pellets of Al-Pa_2O_5 cermet containing 7.3 g of Pa-231 and 10.3 g of Al were canned in aluminum and irradiated in the ORR for 10 hr (nvt = 3.5×10^{18}). After a few hours' cooling, the irradiated slug was transferred to a shielded cell, where the aluminum can was mechanically removed and the pellets were processed for separation and recovery of the Pa-231 and U-232. The process included dissolution of aluminum in 8 M HCl, dissolution of Pa_2O_5 in 8 M HCl—0.6 M HF, selective sorption of uranium on the anion exchange resin column, and elution of uranium product into 0.5 M HCl. Trace protactinium and uranium in the aluminum solution were sorbed on an anion exchange resin.

The time interval between irradiation and processing must be chosen to allow most of the Pa-232 (1.32 day half-life) to decay to U-232 but to limit the amount of Pa-233 (27.4 day half-life) that decays to U-233. A protactinium-uranium separation made after 45 hr decay produced 5 mg of U-232 containing 130 ppm U-233, 90 ppm U-235, and 12,200 ppm U-238. The U-235 and U-238 contamination came from the Pa_2O_5, which contained about 8 ppm of natural uranium. A second Pa-U separation made 93 hr after the irradiation produced an additional 1.8 mg U-232, whose isotopic purity has not yet been determined. Both uranium products require additional purification from protactinium and iron, for which a tertiary amine extraction method is being developed.

FISSION PRODUCT RECOVERY

Preliminary tests showed that o-phenyl-phenol has greater Cs vs. Na selectivity and better phase separation behavior than p-dodecyl-phenol previously tested and may be suitable for recovering cesium from adjusted Purex 1WW waste solution. Greater than 99% of the cesium was extracted from tartrate-complexed simulated high acid 1WW solution, adjusted with

caustic to pH ~12, in a five-stage batch countercurrent test (O/A = 3) with 1 M o-phenyl-phenol in xylene. The cesium was quantitatively stripped in a single contact with 0.1 vol of 0.1 N HNO_3, resulting in an overall Cs/Na separation factor of ~10^3 and a cesium concentration factor of ~3. Strontium, Eu, Ru, and Zr-Nb were not appreciably extracted in separate batch tests.

In a single laboratory test, a double precipitation from synthetic Purex 1WW waste solution recovered 75-80% of the strontium with <1% each of the original Fe^{3+}, Al^{3+}, Na^+, and $SO_4^=$. Sulfate was removed by precipitation after addition of excess ferric ion and increase of the nitric acid concentration to ~65 wt % by the addition of ~90% nitric acid. Strontium was precipitated from the almost pure nitrate system by concentrating the filtrate from the sulfate precipitation step to a boiling point of ~135°C and diluting the residue with ~~fuming~~ nitric acid to >85 wt % nitric acid. The strontium in the nitrate precipitate was dissolved in 1 M HNO_3 solution to provide ion exchange feed solution.

The strontium yield probably could be increased from 75-80% to 80-90% by minimizing the volume of >85% nitric acid from which the nitrates were precipitated since extrapolated solubility data of the $Sr(NO_3)_2$-HNO_3-H_2O system indicated that >15% of the 20-25% unrecovered strontium had dissolved in the large volume (1.7 liters) of >85% HNO_3 solution.

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