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ABSTRACT

Process development studies are reported on Power Reactor Fuel Processing (Studies with Irradiated Uranium Carbides, Dissolution of Sintered UO_2 - PuO_2 Pellets, Shear and Leach Studies, Solvent Extraction Studies, and Protactinium Adsorption Studies); Thorium Utilization Program: The Kilorod Facility (Solvent Extraction Purification of U^{233} and Rod Fabrication); Waste Disposal Development (Pot Calcination of High-Level-Waste, Fixation of High Level Waste in Glass, Low-Level Waste Treatment: Scavenging-Precipitation--Ion Exchange, and Low-Level Waste Treatment: Foam Separation); Chemistry Research (Pa Chemistry, Equipment Decontamination, and Thorium Oxide Studies); Transuranium Element Production (Chemical Process Development: Preparation of PuO_2 for HFIR Target Prototypes, Separation of Americium from Curium at High Alpha Levels, Analytical Chemistry Development: Direct, Nondestructive Method for the Determination of Californium²⁵², TRU Processing Engineering Studies, and TRU Corrosion Studies); and Isotope Development Program (Curium²⁴² Preparation and Recovery of Fission Products).

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POWER REACTOR FUEL PROCESSING

Studies with Irradiated Uranium Carbides. -- Low-level irradiation of uranium monocarbide to about 500 Mwd/ton in a niobium can with an estimated surface temperature of 800°C produced a marked change in the hydrolysis properties of the material. The irradiated carbide when reacted with water at 80°C yielded 96 ml (STP) of gas per gram of sample, consisting of 28% hydrogen, 67% methane, 3% ethane, 1.0% higher alkanes and 0.8% unsaturates. In contrast, unirradiated pellets of uranium monocarbide from the same rods as the irradiated specimens yielded 90 ml (STP) of gas per gram of carbide; the gas consisted of 9% hydrogen, 88% methane, 2% ethane, 0.6% higher alkanes, and 0.2% unsaturates. Only 86% of the carbon present in the specimens before irradiation was found in the gas phase, compared with 98% for the irradiated sample. The non-volatile residue dissolved completely in 6 N HCl, yielding a solution of tetravalent uranium and fission products. Heating unirradiated specimens in a niobium can for three weeks (the length of the irradiation period) at 800°C had no effect on the hydrolysis products, nor did heating other samples of UC to 2000°C. This indicates that the effect observed with the irradiated specimens is not thermally induced.

Dissolution of Sintered UO₂-PuO₂ Pellets. -- Unirradiated fuel pellets containing 80% UO₂--20% PuO₂ (81% of theoretical density, prepared by mechanical blend, fired at 1600°C for 48 hr in an N₂--6% H₂ atmosphere) were dissolved completely in 14, 7, and 3 N HNO₃ in 25, 100, and 180 min, respectively. Similar material, 92% of theoretical density, required 360 min to dissolve in 10 N HNO₃. This high-density pellet contained 82.2% UO₂--17.8% PuO₂ and was coprecipitated and fired under similar

conditions for 16 hr. In each experiment, the ratio of uranium to plutonium in the solution was constant throughout the dissolution.

Shear and Leach Studies. -- Bench-scale leaching tests on sheared stainless-steel-clad UO_2 and ThO_2-UO_2 were continued. In one run, 1-in. sheared lengths of stainless-steel-clad ThO_2-UO_2 suspended in a basket in a recirculating leacher but without sparging were 90% leached in the same length of time (7 hr) as required for 1/2-in. sheared lengths in a sparged system. This result was unexpected, since a much greater surface area was available in the latter system.

A UO_2 batch-leaching run in which 1-1/2-in. sheared lengths of stainless-steel-clad UO_2 were leached in a 10-liter recirculating batch of 7 M HNO_3 showed that UO_2 dissolution was complete in only 1.3 hr. No effort was made to air sparge the UO_2 fines that escaped the perforated basket and collected in the bottom of the equipment.

Solvent Extraction Studies. -- The addition of fluoride to a nitric acid solution of protactinium greatly decreased the extraction of the protactinium with tributyl phosphate. The distribution coefficient of protactinium tracer between 5 M HNO_3 and 30% TBP was 4.0 ± 0.2 , with about 0.5% unextractable. As fluoride is added to the system the scatter of the data increases; however, there is an indication that the distribution coefficient decreases and the percent of niobium which is unextractable increases. The addition of 0.001, 0.01, and 0.1 M HF decreased the distribution coefficient to about 3.3, 0.45, and 0.003, respectively. The corresponding amounts of the protactinium which were unextractable were about 1.3, 36, and 63% of the total protactinium originally present.

Countercurrent batch-extraction experiments with protactinium tracer resulted in the simultaneous extraction of all the uranium and thorium

and about 90% of the protactinium with 30% TBP in n-dodecane from a system using only nitric acid as the salting agent. Decontamination factors were 7, 50, and 250, respectively, from niobium, ruthenium, and rare earth fission products. There was no separation from zirconium.

Protactinium Adsorption Studies. -- The distribution coefficient of tracer Pa²³³ on unfired Vycor from solutions containing 4 to 100 g of thorium per liter and 0.5 to 12 M HNO₃ varied directly with the nitric acid concentration. The expression for the distribution coefficient (y) from solutions containing 4 g of thorium per liter and X M HNO₃ is $y = (388 \pm 10)X + 260 \pm 75$. At 50 g of thorium per liter, $y = (318 \pm 15)X + 342 \pm 106$; and at 100 g of thorium per liter, $y = (259 \pm 5)X + 233 \pm 36$. The amount of unadsorbable protactinium ranged from a maximum of about 4% in 1 M HNO₃ to a minimum of about 0.6% in 12 M HNO₃; from 6 to 12 M HNO₃, the percentage of unadsorbable protactinium increased with increasing thorium concentration but varied only from 1.2 to 0.6%.

THORIUM UTILIZATION PROGRAM: THE KILOROD FACILITY

The Kilorod Facility is being used to prepare about a thousand Zircaloy-2-clad fuel rods containing 97 wt % ThO₂--3 wt % UO₂ for a zero-power criticality experiment at Brookhaven National Laboratory.

Solvent Extraction Purification of U²³³. -- Separation of 4.8 kg of U²³³ from U²³² daughters was accomplished in an Interim-23 flowsheet using a feed of 4.0 g/liter U²³³ and 230 g/liter Th as nitrates (2.6 M H⁺). The feed was contacted in the lower section of a compound extraction-scrub pulse column with solvent (2.5% di-sec-butyl phenylphosphonate in diethylbenzene) and scrubbed in the upper section of the column with an

"acid-deficient" solution (0.8 M Al, 0.4 M acid deficient). The uranium was stripped with water to 10 g/liter in a second pulse column, and concentrated by evaporation to about 120 g/liter for use in the sol-gel process cubicle.

Product from the run met all specifications required for the sol-gel process: (1) decontamination factor (DF) for gross gamma, 230 (specification 100), (2) DF for thorium, 3×10^3 (specification 1×10^3), and (3) product NO_3/U 2.42 (specification, less than 2.50). Total non-recoverable uranium losses were 0.2%.

Sol-Gel Operations -- Twelve cold (thoria-depleted urania) and four hot ($\text{ThO}_2\text{-U}^{233}\text{O}_2$) sols were prepared by blending uranyl nitrate and thoria (prepared by steam denitration of thorium nitrate crystals) at 85°C to an added NO_3^-/Th mole ratio of 0.077. After digestion at 85 to 90°C with circulation for 1 hr, sufficient NH_4OH was added to provide a 0.017 $\text{NH}_4\text{OH}/\text{ThO}_2$ mole ratio. The sol was evaporated to dryness at 85 to 90°C and then fired at 1150°C for 4 to 8 hr in an Ar--4% H_2 atmosphere. Crucibles containing the mixed oxide were removed from the calcining furnace during cold runs at various temperatures ranging from 100 to 350°C to determine the effect of removal temperature on the oxidation state of the product. Equipment performance, not optimum product quality, was the goal in the first five cold runs.

The product U/U+Th ratio met specifications (3.00 ± 0.03) in all cold runs after run 5. During cold runs 6 through 9 the product O/U ratio was 2.104, 2.030, 2.040, and 2.085, respectively (specification ≤ 2.04). The above specifications can be met during the Kilorod program, but the current oxide gas release specification (≤ 0.01 cc/g) probably cannot

(average gas release to 1200°C ranged from 0.020 to 0.11 cc/g during runs 5 through 9). Pulling oxide from the calciner at 350°C (necessary if the 24-hr overall calcining cycle is to be achieved) did not appreciably alter the product oxidation state. The product size distribution was satisfactory in all cold runs.

The U/U+Th ratio for the first hot sol was 2.98. Subsequent batches have not been fully analyzed.

Rod Fabrication. -- Compacted densities of about 90% of theoretical were obtained with the cold oxide in two size fractions --- 55% -6 +16 mesh, and 45% fines (composite of all material below 16 mesh). This density was also obtained on the first two rods loaded with $\text{ThO}_2\text{-U}^{233}\text{O}_2$. Density within a rod appeared to be within the $\pm 2\%$ specification.

WASTE DISPOSAL DEVELOPMENT

Pot Calcination of High-Level-Waste. -- Three continuous evaporator pot-calcination tests using FTW-65 Purex waste (simulated formaldehyde-treated waste of a composition expected from Hanford in 1965) were completed. The average feed rates varied from 31 to 49 liters/hr, and the maximum hourly rates from 125 to 190 liters/hr. The average feed rate is a function of the concentration in the evaporator and the degree of filling of the calciner pot. The calcined solids had densities of 1.23 to 1.30. The residual nitrate composition for the calcined solids varied from 100 to 400 ppm and decreased with time that the calcined solid was above 600°C. The sulfate retention in the calciner was high: only 0.3 to 2% of the sulfate was lost. This loss is attributed to entrainment since similar values were obtained for nonvolatile ions such as iron and sodium. A catch tank, tested as a method of isolating

mercury, collected only 27% of the mercury. The amount of mercury caught was proportional to the amount in the last fraction of the calciner condensate. A ruthenium decontamination factor of 3×10^3 (using nonradioactive ruthenium) was obtained in test R-72 across the continuous evaporator. In this test an impingement plate de-entrainer was used.

Fixation of High-Level Waste in Glass. -- Simulated Hanford FTW-65 waste was evaporated to 1/4 of original volume without precipitation of solids. Experimental phosphate glassy melts were made by addition of phosphate, lithium, sodium, and calcium to FTW-65. Generally, these melts were too soluble in water or lost SO_3 readily, but a few appeared to be insoluble and were relatively stable at high temperatures. Substitution of lithium for part of the added sodium resulted in solids that melted from 50 to 100°C lower than the corresponding sodium compounds and that were less soluble in water. A semicontinuous, semiengineering-scale experiment with the lithium glass was successful, although excessive solids entrainment caused an operating problem. The product contained 45% waste oxides and represented a volume reduction factor of 30.3. Sulfate loss was less than 2% and 24% of the ruthenium volatilized. An electrical resistance probe for sensing levels of solution, calcine, and melt in the pot performed satisfactorily.

Imposition of an electrical potential decreased the time necessary for removal of more than 99% of the mercury in a copper column from FTW-65 waste solution from about 25 to 12 min or less. More current was required than the minimum to remove the mercury in order to prevent dissolution of excess copper.

Exposure of type 304L stainless steel specimens at 300°C in bedded salt (NaCl) containing naturally occurring impurities showed corrosion rates of about 0.03 mil/month. Type 304 stainless steel and titanium 45A, both coupled and uncoupled to each other, showed low rates for exposure in refluxing FTW-65 waste solution and vapors; the maximum rate was on the coupled 304L and amounted to 0.3 mil/month with no local attack.

The thermal conductivity characteristics of powdered MgO, Al₂O₃, and ZrO₂ were investigated as a function of void space and temperature. Over the temperature range studied (200 to 1500°F), the thermal conductivity increased quadratically with the temperature.

Low-Level Waste Treatment: Scavenging-Precipitation--Ion Exchange. --

The phosphate concentration of ORNL low level waste was successfully reduced from ≥ 3 ppm of phosphate to 0.2 to 0.5 ppm during treatment of greater than 8500 volumes of waste with one bed volume of alumina. This treatment permitted more-quantitative precipitation of calcium carbonate in the scavenging-precipitation clarifier, i.e., reduction in hardness of clarified product from 70 ppm to less than 10 ppm. A significant but unknown fraction of the ruthenium was also adsorbed on the alumina.

Low-Level Waste Treatment: Foam Separation. Initial tests of a two-step process for decontamination of low-level waste, consisting of sludge column precipitation of calcium carbonate and magnesium hydroxide followed by a countercurrent foam column for strontium removal, were performed with Sr⁸⁵-spiked ORNL tap water and ORNL low-level process waste water in laboratory-scale equipment. With tap water as feed, strontium decontamination factors in the foam column and in the overall process were 2 to greater than 400 and 320 to greater than 15,000, respectively, depending

on operating conditions. With process waste water, foam column and overall strontium decontamination factors were 11 to 16 and about 250, respectively, which are close to those obtained with tap water under the same operating conditions. Also tested with simulated and actual ORNL low level process waste was a one-step process which involves precipitation of calcium as orthophosphate in a small mixer and then feeding solution and solids to a foam column where flotation and foam separation occurs. Strontium decontamination factors with simulated and actual ORNL waste water were, respectively, in excess of 1000 and 200 to 300.

Studies were performed on the biological degradation in ORNL process waste water of the alkylbenzenesulfonate already present in the waste water (primarily from Fab, a household detergent) of the alkylbenzenesulfonate in Fab, and of the branched chain sodium dodecylbenzenesulfonate of Trepolate F-95. After 2 to 3 weeks the respective amounts of degradation from existing bacteria were 20, 25 to 30, and 60 to 65%.

A 24-in.-diam foam column was operated with liquid feed introduced through tubes on a 5-in. triangular spacing; channeling was observed for 10 to 30 in. below the feed tubes. Twenty-mesh screens across the column cross sections were not effective liquid distributors since they accumulated liquid, which dumped through periodically. Good drainage of foam was obtained in a horizontal drainage section. The experimentally measured foam densities agreed well with a foam drainage model. Air-operated sonic whistles condensed 0.05 to 0.10 ft³ of foam per standard cubic foot of operating air. The experimentally observed concentrations for a three-stage surfactant recovery system agreed well with concentrations calculated by using a distribution coefficient of 7×10^{-4} (moles/cm²)/(moles/cc) for the docecylbenzenesulfonate.

CHEMISTRY RESEARCH

Pa Chemistry. -- Additional protactinium distribution measurements between 5 N sulfuric acid and 0.03 N trilauryl amine in diethylbenzene confirmed that there is a change in distribution behavior as the protactinium concentration is increased. For aqueous-phase Pa²³¹ concentrations, less than approximately 2×10^5 alpha counts $\text{min}^{-1} \text{ml}^{-1}$, the concentration in the organic solvent was proportional to the 1.3 power of the aqueous phase concentration. At higher concentrations, the protactinium concentration in the solvent was proportional to the 3.2 power of the aqueous-phase concentration. This behavior is similar to that observed in concentration dependence between 5 N sulfuric acid and Dowex 1 resin, except that the change was even larger in the case of the resin.

These observations are generally consistent with a reversible polymerization of protactinium in one or both phases, with both the polymer and the simpler species being extracted, but the polymer being more extractable. The polymer is small, perhaps a hexamer, and it is formed reversibly with respect to changes in the concentration of both protactinium and sulfuric acid. Polymer formation is inhibited by sulfuric acid, with the dependence on acid concentration being second power.

Equipment Decontamination. -- A solution of 0.5 M oxalic acid with 0.1 M F^- and 0.2 M H_2O_2 at 95°C, which was an effective decontaminant for stainless steels contaminated in helium at 500° to 650°C, produced no stress-corrosion cracking on U-bends of types 304, 316, 321, and 347 stainless steels in 22.5 hr. The solution strength was restored every hour by the addition of 0.1 M $\text{H}_2\text{C}_2\text{O}_4$ + 0.1 M H_2O_2 .

Several samples of contaminated stainless steel from loop 2 of the Oak Ridge Reactor were successfully decontaminated. Barium-140 was a prominent contaminant of all samples. Those samples which had been exposed to contaminated helium at 540 to 600°C were also contaminated with Ru, Zr, Nb, and Ce. One such sample had a decontamination factor (DF) of only 7 in 3 hr at 95°C in the noncorrosive pH-4 solution of oxalate-citrate-peroxide. In 3 additional hours at 95°C in a more corrosive solution of 0.5 M $H_2C_2O_4$ --0.1 M NaF--0.2 M H_2O_2 , the total DF was 54. The DF was 84 on another sample, contaminated chiefly with Ba^{140} at 270°C, in 1 hr at 95°C in the pH-4 solution. In 3 N HNO_3 in 1 hr at 95°C the DF was 128. A graphite sample exposed to helium at 600°C resisted decontamination, probably due to its porosity (DF = 1.6 in 1 hr at 95°C in pH-4 solution).

The decontamination of Zircaloy-2 should be very rapid and complete in solutions of oxalic acid containing fluoride and hydrogen peroxide. The uniform dissolution rate in 0.5 M $H_2C_2O_4$ --0.1 M NaF--0.2 M H_2O_2 at 25°C was 3.4 mils/hr, without pitting or gas formation. With 0.05 M $H_2C_2O_4$ --0.05 M NaF--0.07 M H_2O_2 , the rate was 1.1 mil/hr. The Zircaloy surfaces were very bright and clean.

Thorium Oxide Studies. -- Stable sols of crystalline (calcined at 500 to 850°C) thorium oxide suspended in dilute nitric acid were studied by photomicrography, x-ray, and a variety of physical chemistry methods. The colloidal particles of these sols appear to have three levels of organization: single crystallites; tightly bonded low-number multiples of crystallites; and elongated, weakly associated agglomerates of crystallites. The single crystallites are nearly centrosymmetric and

range in size from 70 to 250 A, depending on calcination temperature. Tightly bonded multiples of crystallites are produced by the higher calcination temperatures and have axial ratios near 2. High-dilution viscometry discloses the hydrodynamically effective particles to have axial ratios of 6.5 to 8.5 at pH 2.00, eccentricity decreasing with particle concentration at constant pH. This behavior, as well as exaggerated non-Newtonian flow properties at solids volume fractions exceeding 0.05, are believed associated with reversible agglomerate structures rather than with the crystallites themselves.

Electrophoretic measurements for thoria in pH 2.00 nitric acid showed a three-day aging effect and high value of the apparent zeta potential, which increased to a value of 90 mv over the three-day period. This is higher than results found in published data. However, first-order electroviscous effects are expected to be negligible at the high particle eccentricity and high ionic strength used in the present experiments.

TRANSURANIUM ELEMENT PRODUCTION

Chemical Process Development: Preparation of PuO₂ for HFIR Target Prototypes. -- Eighty-five grams of high-fired PuO₂ containing about 75 g of Pu²³⁹ were prepared for use in prototype HFIR targets. At 150°C, Pu(OH)₄ was precipitated and dried. The hydroxide was ground through a 70-mesh screen, and the +325 mesh particles were fired in air at 1200°C to produce glassy solid particles with a density of 10.99 (96% of theoretical). Particle size of the fired oxide is as follows:

<u>Screen Size</u>	<u>Weight %</u>
-70 +140	43.5
-140 +170	16.3
-170 +200	8.6
-200 +325	24.3
-325	7.3

This material will be mixed with aluminum powder (-325 mesh) and pressed into cermets. With PuO_2 particles of this size, the aluminum phase should be continuous, and conductivity will be satisfactory for irradiation in high neutron fluxes.

Separation of Americium from Curium at High Alpha Levels. -- Separation of americium from curium by chromatographic elution from a cation exchange column with ammonium α -hydroxyisobutyrate at activity levels of about 50 w/liter was attempted. Four milligrams of Cm^{242} ~~was~~ loaded on a 30-ml column of Dowex 50-8X (-400 mesh) resin at 80°C from a solution containing americium and curium. The loaded column was eluted with 0.4 M ammonium α -hydroxyisobutyrate at pH 4.2 at a flow rate of 0.4 ml $\text{cm}^{-2} \text{min}^{-1}$. Extensive gassing problems were encountered, with gas pockets up to 1 in. long developing. However, in one of the runs, a 70% curium recovery with a 14% americium carryover was obtained. The column was modified with a spring-loading device that kept the resin bed compacted. Gas pockets and channeling were prevented despite large amounts of radiolytic gas, and a curium recovery of 90% with a 10% americium carryover was achieved. A separation factor of 1.4 was obtained, and the system behaved well chemically.

Analytical Chemistry Development: Direct, Nondestructive Method for the Determination of Californium²⁵². -- The prompt gamma rays accompanying

the spontaneous fission of Cf^{252} offer the interesting possibility of a direct, nondestructive method for its determination. Californium-252 has an alpha half-life of 2.55 yr and a spontaneous fission half-life of 66 yr. The gamma-ray spectrum in coincidence with spontaneous fission extends to about 5 Mev. Each fission event yields an average of 10 to 12 gamma rays. Discriminatory gamma scintillation counting has been found to provide a sensitivity of at least 0.01 nanogram of Cf^{252} . The method is applicable to the direct determination of Cf^{252} in the presence of other actinide elements and gross fission products. Details will be given in a forthcoming report.

TRU Processing Engineering Studies. -- Performance of a manipulator-booting-seal extractor was satisfactory; however, application of talcum powder to the rubber seal surfaces before installation was necessary to prevent excessive friction during extraction. From tests in the cell mockup, alterations in the cell lighting system were recommended to give more uniform intensities and easier remote maintenance. A tantalum-to-Hastelloy rolled joint was tested with temperature cycling from ambient to 285°C . The joint appears acceptable from the intended uses, with no detectable leakage for 15 psig below 200°C .

TRU Corrosion Studies. -- The corrosion rate for grade-11 zirconium was very slightly accelerated from a 1000-hr rate of 1.7 mils/month to a 1000-hr rate of 2.1 mils/month when 50 ppm of ferric ions as $\text{FeCl}_3 \cdot \text{H}_2\text{O}$ were added to boiling and oxygenated 6 M HCl solution. The hydrogen content of the specimen in the absence of ferric ions was 790 ppm; this value increased to 1300 ppm when ferric ions were present. Both specimens underwent brittle fracture during bending upon completion of the tests.

Solution and vapor specimens of Hastelloy C corroded at constant rates of 0.3 and 1.0 mil/month, respectively, after a 1000-hr exposure to oxygenated 10 M LiCl--0.1 M HCl solution at 43°C. Both specimens experienced preferential attack in heat-affected zones adjacent to welds, with the heavier attack, up to 10 mils in depth, occurring on the vapor-exposed specimens.

ISOTOPE DEVELOPMENT PROGRAM

Curium²⁴² Preparation. -- About 20 mg of Cm²⁴² was recovered from an irradiated Am²⁴¹ target in Building 3019 analytical hot cells by the LiCl-anion exchange method. Curium product analysis by gamma-ray spectrometry showed Ru¹⁰³ to be the only gamma-emitting fission product present. Gross fission product decontamination was greater than 10⁴. The 20 mg of Cm²⁴² product produced 2.5 r/hr of gamma radiation; however, this activity was completely attenuated with 1 in. of steel.

The recovered Cm²⁴² is being used in Building 3508 for the evaluation of the extent of acid depletion by radiolysis of chloride solutions, separation processes, and physical handling of 10-20 w/liter solutions.

The principal effect of alpha radiation in Tramex feed is a loss of acid by radiolysis. Attempts were made to stabilize the acidity of the feed solution by the addition of materials that liberate HCl by hydrolysis. Hydrazine dihydrochloride and hydroxylamine hydrochloride were investigated. Both hydrolyze to give approximately the required concentration of HCl. However, in the presence of high alpha activity they liberate acid at a faster rate than acid is destroyed, and a net hydrogen ion increase results. Therefore, addition of an excess of these

materials does not appear to be a satisfactory way of stabilizing the acidity of Tramex feed for long term storage.

Bubbling dry helium gas containing small amounts of HCl through Tramex feed was evaluated as a possible method of acid adjustment. Equilibrium acid concentrations were determined for 10 and 11 M LiCl solutions contacted with dry helium gas containing HCl at a partial pressure of 23.5 mm. Acid concentrations of 0.31, 0.20, and 0.14 M were obtained at 70, 100, and 120°C, respectively, for 10 M LiCl, and acid concentrations of 0.18, 0.11, and 0.08 M were obtained at 70, 100, and 120°C, respectively, for 11 M LiCl. Equilibrium acid values were greater than 0.1 M; therefore, this procedure appears to be of dubious value because acid must be less than 0.1 M for satisfactory actinide extraction, and the HCl partial pressure cannot be reduced appreciably below 23 mm and still replenish acid lost by radiolysis at a reasonable rate of gas flow.

Another method that makes use of an electrochemical pH-Stat developed by Posey, Morozumi, and Kelly of the Chemistry Division was investigated for replenishing acid. In radioactive solution, the electrode was seriously poisoned. It is doubtful if electrodes satisfactory for service in Tramex feed at 10 w/liter can be found.

Recovery of Fission Products. -- In a continuous countercurrent demonstration run in miniature mixer-settler equipment, more than 99.3% of the cesium was recovered from tartrate-complexed simulated Purex 1WW waste (pH 12.7) with 1 M 4-sec-butyl-2-(α -methylbenzyl)phenol (BAMBP) in Amsco 125-82. The extract was scrubbed with 0.002 M sodium tartrate and stripped with 0.1 M HNO₃. Flow ratios, aqueous feed/organic/scrub/strip,

were 2/1/0.2/0.05. No apparent change in performance was noted through 26 hr of operation, equivalent to 23 complete cycles of the solvent through the system.

Effective extraction of technetium from simulated Hanford tank-waste supernatant was obtained with quaternary amine extractants. The extraction coefficient (1/1 phase ratio) with 0.17 M Aliquat 336 in 94% diisopropylbenzene--6% tridecanol was 75 at pH 9 and 130 at pH 13.



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- 61-75. DTIE
76. Research and Development, ORO