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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

MAY 1958

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May 1958

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Oak Ridge, Tennessee
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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

May 1958

This Status and Progress Report summarizes somewhat more than one-half the activities of the Laboratory. Some of the topics are included every month, but many of them are reported on a bimonthly schedule. Program 4700 is covered in a separate report.

PROGRAM 2000 — SPECIAL NUCLEAR MATERIALS

Waste Metal Recovery. — A total of 8 tons of bomb-test sand was processed for recovery of its 76 g of plutonium and 83 g of uranium. The leaching and dissolving operation removed 95% of the plutonium contained in the sand. The product of a continuous pulsed column solvent extraction operation contained a siliceous-organic material which caused erratic evaporator operation and the subsequent loss of plutonium. When batch extraction and stripping were substituted for the continuous column operation, plutonium recovery was 75%.

The second Paducah ash program was initiated. A total of 3 tons of fluorinator ash residue, containing 236 g of Np^{237} and ~ 1.8 tons of uranium, was dissolved, and neptunium and uranium recoveries from this material averaged 99.8 and 99.7%, respectively. (AEC Activity 2350)

Excer Process. — A sulfate uranium-processing flowsheet was demonstrated in a series of runs. Uranyl sulfate, recovered from ore leach liquor, was reduced to uranous sulfate by metallic iron. The uranous sulfate was separated from iron by anion exchange. It was converted to uranous chloride in a 2-in.-dia continuous ion exchange contactor containing 50- to 100-mesh Dowex 50 cation exchange resin by elution with 6 M HCl. The production rate was 11 lb of uranium per hour per square foot of column area. The HCl consumption was about 9 lb per pound of uranium. About 9% of the uranium appeared in the waste, owing to oxidation of U(IV) to U(VI), but this can be conveniently recycled in the sulfate flowsheet.

Twenty-two pounds of uranium, as uranyl nitrate, was converted to uranyl chloride by continuous ion exchange in a 2-in.-dia contactor and reduced to uranous chloride in a 9- by 10-in. Ionics membrane electrolytic cell. The product was precipitated as uranous oxalate, redissolved in ammonium oxalate, coprecipitated with thorium oxalate, and fired to make mixed ThO_2-UO_2 slurry for use in homogeneous reactor blanket studies. (AEC Activity 2702)

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Fluorox Process. — A rate constant, k, of 2.05 hr^{-1} at 800°C was calculated for the reaction $2UF_4 + O_2 \longrightarrow UO_2F_2 + UF_6$ from data obtained in operation of the 3-in.-dia fluidized-bed reactor. This value is consistent with the values obtained in laboratory studies. The new 4-in.-dia fluidized-bed reactor was successfully operated at 825°C for 12 hr; 10.5 lb of UF_6 was collected. (AEC Activity 2702)

PROGRAM 4000 — REACTOR DEVELOPMENT

HOMOGENEOUS REACTOR PROJECT

Homogeneous Reactor Test. — Before the beginning of tests to determine the most feasible means for future operation of the HRT, several more attempts were made to view the core leak site. A 21-ft borescope was inserted through the upper screens, but illumination at the core wall was insufficient to permit inspection. A remotely operated mirror was then inserted into the blanket to allow the previously unexamined southeast quadrant of the core to be observed by periscope. As in earlier observations of the southwest quadrant, only a smooth, white external core surface was visible in the mirror. Thus the core "hole" has not yet been located, although the elevation has been determined, as was previously reported.

During the shutdown for viewing, the fuel letdown valve was replaced by use of remotely operated tools and a lead-shielded dry-maintenance personnel platform. Also, the steam generating capacity of the blanket heat exchanger was increased by increasing the size of the feed-water and steam withdrawal valves.

Run 15 commenced on May 9, with both systems filled with condensate and with heating supplied externally. The purpose of the run was to determine a satisfactory method of maintaining pressurization and liquid-level control. At 150°C the pressure drops through the core and blanket vessels were similar enough to permit control within the range of the pressurizer level elements; however, the lower density at 280°C caused the blanket pressurizer level to be above the control region.

Pressurizer control achieved in run 15 with a perforated diaphragm in the relief line between core and blanket pressurizers was unsatisfactory, so the line was closed with a solid blank. Run 16 was then performed at 150°C and 1800 psi, with dilute UO_2SO_4 in the core, to discover whether two-region separation could be maintained by completely filling the blanket pressurizer and continuously purging 6 lb/min of D_2O from the blanket into the core.

System pressurization and fuel pressurizer level control were found satisfactory; however, blanket samples indicated that uranium from the core was mixing with the blanket in spite of the purge, and operation was suspended to rinse the blanket system and return the transferred uranium to the fuel dump tank. After further exploration of pressurizer level control methods, the blanket pressurizer was refilled to repeat the mixing experiment and allow it to proceed to equilibrium. The experiment was interrupted because of a broken blanket-sampler-valve stem but will be resumed when repairs have been completed. (AEC Activity 4103.3)

Fuel Processing. -- Silvered Yorkmesh stainless steel packing continued to remove iodine from a steam-oxygen mixture (1% O_2) containing traces of iodine after 60% of the silver had reacted with iodine. However, when the oxygen content was increased to 10% at a temperature of 150°C, oxidation and consequent destruction of the Yorkmesh occurred; silvered Yorkmesh containing no iodine but subjected to the oxygen-enriched vapor was not affected.

Electrolysis of simulated HRT dump-tank fuel solution with a rotating copper cathode resulted in the deposition of copper containing about 1% of the total nickel with only slight reduction of uranyl to uranous ion. With stationary platinum electrodes and mechanical stirring in a single-compartment cell, about 50% reduction to uranous ion was observed, with 10% of the nickel being deposited from solutions which contained no copper. (AEC Activity 4103.1)

Thorium Oxide Slurry Development. -- Following modification of the production equipment of the thorium oxide pilot plant, 250 lb of 1600°C fired thorium oxide, classified to $< 5\,\mu$ and recalcined at 650°C, was prepared; 37 lb of oxide similarly treated but classified to < 7 and $> 3\,\mu$ was also prepared.

In thorium oxide preparation studies, thorium-uranium oxide (U/Th = 0.05) was prepared by bubbling CO_2 through an aqueous suspension of ThO_2 and $UO_3 \cdot H_2O$ and firing the resulting solid at 1000°C for 18 hr. Less than 10% of the uranium was solubilized by subsequent treatment of the material at room temperature with 8 N HNO_3.

The yield stresses of slurries containing thorium oxide (geometric mean particle size, $1\,\mu$) at a concentration of 1000 g of Th per kg of H_2O were 0.85 lb/ft^2 for oxide fired at 650°C and 0.25 lb/ft^2 for material fired at 1200 to 1600°C. The coefficients of rigidity were 3 to 5 centipoises and were relatively insensitive to the firing temperature above 800°C.

A slurry of 1000°C fired thorium-uranium oxide (U/Th = 0.05) containing no gas-recombination catalyst, at a concentration of 500 g of Th per kg of D_2O, generated radiolytic gas pressures of 170 to 200 psi at 270 to 300°C when irradiated in the LITR. The apparent over-all activation energy for gas recombination in a slurry of 650°C fired thorium-uranium oxide containing 2.7% U^{235}, at a concentration of 500 g of Th per kg of D_2O, under irradiation in the Graphite Reactor was 16.2 kcal per mole of H_2. The same value was obtained previously with a light-water suspension of the same preparation. (AEC Activity 4103.1)

Gaseous-Fission-Product Disposal. — Studies of the adsorption of trace quantities of krypton from a flowing stream of oxygen by silica gels, alkaline-earth silicates, and molecular sieves at temperatures up to 300°C showed the sieves to be the most efficient noncharcoal adsorbers. The order of efficiency at temperatures higher than 25°C is the same as at 25°C, namely, molecular sieve 5A > 13X > 10X, with the differences disappearing as 300°C is approached. Static adsorption isotherms for krypton on Columbia G charcoal, molecular sieve 5A, and silica gel grade 35 were obtained at pressures ranging from 0.01 to 0.6 mm by tracer techniques. The adsorptive capacities of these materials (mg of krypton per g of adsorbent) at 0.1 mm pressure are 0.0296, 0.0062, and 0.0028, respectively, at 28°C. These values are about a factor of 5 lower than those obtained by gravimetric methods of questionable accuracy. (AEC Activity 4103.1A)

Mathematics and Computation: Homogeneous Reactor Development. — The standard two-group, two-region Oracle code for spherical reactors was modified to include fissions in the fast group. A first-flight correction was added to an Oracle code which calculates critical concentrations in one-region reactors. A stress-analysis calculation for transition joints between dissimilar metals and a numerical integration procedure for a first-order differential equation were programed for the IBM-704. (AEC Activity 4103.1)

Reactor Analysis. — The accuracy with which the breeding ratio of the HRE-3 could be determined after a period of reactor operation was investigated. Inaccuracies in the core U^{233} inventory and in the blanket U^{233} and Pa^{233} inventories appear to be the major sources of error. Appreciable errors could result from attempting to determine inventories by sampling the reactor contents.

The influence of the core inlet-outlet line on the flux distribution in the HRE-3 was examined by use of a reactor code which treats two space variables. The ratio of local to average flux was increased somewhat in the vicinity of the inlet, but the breeding ratio was reduced only 0.3%.

The reduction in the HRE-3 breeding ratio resulting from neutron absorptions in the core inlet line was examined for spherical and cylindrical cores; it was found to be less than 0.5% if the pipe does not extend more than one-fourth the way to the bottom of the core. The power density at the end of the pipe would be 57 kw/liter if it extended 5 in. into the sphere.

The nuclear characteristics of the HRT were calculated for operation with the same fuel concentration in the core and blanket regions. The critical concentration would be 2.5 g of U^{235} per liter in a clean system at 280°C. About 70% of the power would be generated in the blanket region.

In studies of the safety of the HRT when operated as a one-region reactor, the one-region system appears to be as safe as the two-region system with respect to the effects of possible additions of reactivity.

Pressure and thermal stresses in the wall of the HRT pressure vessel were computed for both one- and two-region reactor operation. The stresses were about the same for the two cases. On the basis of permissible stresses, the power appears to be limited to about 5 Mw if there is no heat removal from the outer surface of the pressure vessel, but it may exceed 10 Mw if the inner- and outer-surface temperatures are equal. (AEC Activity 4103.1)

Homogeneous Reactor Instrumentation. — Calculations were made in which the HRT steam-system valves were resized to permit single-region reactor operation with nominal full-power extractions from the blanket and core heat exchangers, respectively, of 7 and 3 Mw. The two condensate blowdown valves were also decreased in size to reduce the pressure transient experienced during a blowdown operation.

Modification of the reactor control system to facilitate single-region operation was studied. Firm recommendations will be made after preliminary test operation has established the procedures necessary for satisfactory mechanical operation.

A 2-in.-dia television camera with remote camera electronics and designed to go inside the HRT core vessel was received. The camera produced a satisfactory picture of objects at distances greater than 4 in. when they were illuminated with a small lighting turret concentric with the lens. After being tested, the camera, cable, and remote electronic chassis were returned to the manufacturer for repair of minor cable defects and the substitution of more radiation-resistant insulation in the camera head.

An experimental letdown valve was designed which incorporates a 1/8-in.-dia body port and a lower stem guide to provide better plug-to-seat alignment. The guide is ported to avoid fuel-solution stagnation in the stem sealing-bellows compartment. The smaller bore (previous designs were 1/4 in.) permits an increase in the plug-to-seat clearance, which should lessen the possibility of solid particles wedging the plug in the seat. (AEC Activity 4103.1)

HRP Design. — Studies are being made of the changes required for the operation of the HRT as a single-region reactor. An investigation of mixing between the core and blanket high-pressure systems indicates that equilibrium solution concentration, at an optimum mixing rate of about 75 to 100 lb/min, would be reached in about 4 hr. System modifications necessary for single-region operation include interconnection of the fuel and blanket purge-pump

discharge lines, alteration of the steam-system piping to permit independent control of the core and blanket steaming conditions, and addition of an iodine adsorption bed to the blanket catalytic recombiner. Design and procurement for the adsorption bed are complete.

Studies of replacement of the HRT reactor vessel are also being made, including redesign of the vessel, modification of piping to accept the redesigned vessel, and removal of the old vessel and installation of the new. A hydraulic manipulator was designed to permit inspection of the interior of the existing vessel by means of a television camera. A study of the feasibility of dry maintenance of the HRT was completed by the Vitro Corp., two of the concepts presented appearing worthy of further consideration.

Conceptual-design studies for the HRE-3 were discontinued in favor of preliminary design studies for a 1- to 5-Mw experimental, single-region slurry reactor. A list of problems requiring specific development work preliminary to design of such a reactor was prepared. (AEC Activity 4103.1)

HRP Metallurgy. — Beta-quenched ternary Zr—15 wt % Nb—(Mo or Pd) alloys have now been investigated for hardness changes while being aged at a variety of temperatures below 600°C. The addition of either molybdenum or palladium slows down the hardening omega transformation sufficiently to permit welding. The transformation, which in the binary alloys occurs in minutes, was delayed 2 hr by adding 5 wt % Pd, 4 hr by 2 wt % Mo, and more than two weeks by 5 wt % Mo.

While it is possible to fabricate Zircaloy-2 by a variety of schedules and to obtain conventional tensile properties that show no differences between the longitudinal and transverse directions, such material still possesses preferred orientation. The fracture cross sections of such specimens are elliptical rather than round, indicating that the reductions are not uniform in all directions. If the ratio between the percentage reduction in length of the two axes is calculated, a reproducible measure of anisotropy is obtained. Such a ratio was found to be about 13 for commercial material and only 2.5 for material fabricated by improved fabrication procedures.

Subsize impact samples of crystal-bar zirconium and Zircaloy-3a exposed to fissioning uranyl sulfate solution in the in-pile loops showed no change in properties as a result of the exposure. Similar results were obtained previously with Zircaloy-2. (AEC Activity 4103.1)

Laboratory Corrosion Studies. — Cracking in type 347 stainless steel bent pipe, heated internally with 150°C steam and cooled externally with chloride-containing tap water, has been prevented for 5000 hr by placing exposed surfaces under compressive stress by shot-blasting. Cold-formed bends that have not been shot-blasted normally crack during 1000 hr under the same conditions. Wrapping the surface of a cold-formed pipe bend with an aluminum-wire spiral also prevented cracking during a 2000-hr period.

High-nickel alloys such as Incoloy and Nionel were found to be more resistant to stress-corrosion cracking than were the austenitic stainless steels in pH 10.5 water containing 100 ppm chloride at 300°C. However, at pH 2.8 the nickel-base alloys were as susceptible to cracking as were the austenitic stainless steels.

Niobium exhibited a mild pitting attack in uranyl sulfate solutions at 100 and 200°C; at 300°C the metal underwent catastrophic attack. Further testing with Croloy 16-1 stainless steel demonstrated that the alloy is not suitable for use in uranyl sulfate solutions. (AEC Activity 4103.1)

Dynamic Solution Loop Corrosion Tests. — Testing of the mockup of the Zircaloy-2—stainless steel transition and expansion joint used in the HRT reactor vessel was completed. The total exposure of the mockup to solution circulating at 100 to 300°C has been for 9153 hr, including 8600 hr on fuel solution (0.04 m UO_2SO_4, 0.02 m H_2SO_4, 0.005 m $CuSO_4$), 312 thermal cycles between 300 and 100°C, and 518 mechanical deflections (5/8 in.). There was no detectable leakage through the expansion bellows or across the transition joint.

In further investigations of solution stability it was found that 0.04 m UO_2SO_4 containing 0.025 m D_2SO_4 and 0.03 m $CuSO_4$ in heavy water (the same composition as used in HRT run No. 14) was stable at 300°C with up to 750 ppm nickel as nickel sulfate. About 35% of the nickel and 7% of the copper were lost from solution when the nickel concentration was increased to 1700 ppm. When the temperature of the system was lowered to 150°C, the precipitated nickel dissolved, but the precipitated copper did not dissolve.

It was also found that 0.04 m UO_2SO_4 containing 0.025 m D_2SO_4, 0.03 m $CuSO_4$, and 0.04 m $MgSO_4$ in D_2O was stable for 200 hr at 300°C. At 250°C this solution in one test was less corrosive to stainless steel than was the same solution without the magnesium sulfate. Also, the critical velocity on type 347 stainless steel coupons was higher in the presence of magnesium sulfate than in its absence. Longer-term tests must be made, however, to prove that the solution is stable, particularly with additions of nickel sulfate. (AEC Activity 4103.1)

Small-Scale Dynamic Slurry Corrosion Tests. — Relative effects of temperature, gaseous atmosphere, and thoria preparation on the corrosion of several metals in neutral slurry were derived from analysis of results of toroid tests. The normalized attack rate for type 347 stainless steel at 280°C under an oxygen atmosphere was 4 mpy (mils per year). The attack rates on several metals relative to the attack on type 347 stainless steel were as follows: titanium-75A, 0.5; Zircaloy-3A, 0.4; and SA-212-B steel, 8.0. The attack rates at several temperatures relative to the attack at 280°C were as follows: 100°C, 0.14; 150°C, 0.5; 200°C, 0.3; and 250°C, 0.7. Attack rates with hydrogen overpressure were four times as great as those obtained with oxygen overpressure.

The presence of $NaAlO_2$ in aqueous oxygenated thoria slurries resulted in an increased attack on all alloys tested. At a concentration of 1000 g of Th per kg of H_2O, oxygenated slurries with and without $NaAlO_2$ were circulated in toroids for 300 hr at 26 fps at a temperature of 280°C. The presence of 0.14 m $NaAlO_2$ in the slurry (pH 10) increased the attack rates as follows: type 347 stainless steel, from 6 to 30 mpy; titanium-75A, from 2 to 140 mpy; SA-212-B mild steel, from 14 to 40 mpy; and Zircaloy-3A, from 1 to 3 mpy. Lower concentrations of $NaAlO_2$, with lower pH, gave less effect. In a separate test, removal of sodium ion by dialysis reduced the attack on Zircaloy, titanium, and stainless steel. (AEC Activity 4103.1)

Dynamic Slurry Loop Corrosion Tests. — A 719-hr test in the new slurry loop ES was completed. Under an oxygen atmosphere, thoria calcined at 1600°C (1 μ average diameter) was circulated at 280°C at an average concentration of 1150 g of Th per kg of H_2O. Attack on the Zircaloy impeller, titanium pump-scroll inserts, aluminum oxide bearings, and other components was mild. The loop corrosion rate averaged 1 mpy. No attack was noted on titanium or Zircaloy specimens at velocities up to 45 fps. Attack rates (in mpy) of special materials at various velocities were as follows:

Velocity (fps)	Stainless Steel	Stellites	Kennametal K501 (WC in Pt)	Tungsten Carbide	Rexalloy	Aluminum Oxide
9	0.1	1—6	>1200	33	5	31
20	0.5	10—170	>1200	39	180	35
27	2	80—210	>1200	42	190	56

(AEC Activity 4103.1)

In-Pile Solution Corrosion Loops: LITR. — Corrosion rates obtained on Zircaloy-2 core specimens at 235°C in in-pile loop L-4-18 (0.17 m UO_2SO_4, 0.02 m H_2SO_4, 0.07 m $CuSO_4$ in D_2O) were lower than those obtained at 250°C in other loop experiments. This decrease in rate with decreasing temperature agrees qualitatively with autoclave results at 250°C and below.

Zirconium and Zircaloy-2 specimens from L-2-19 (0.17 m UO_2SO_4, 0.10 m H_2SO_4, 0.02 m $CuSO_4$, 0.20 m Li_2SO_4 containing 99.9% Li^7 at 280°C) exhibited corrosion rates, for similar solution power densities, in agreement with those of L-2-14, which employed a solution of 0.17 m UO_2SO_4, 0.4 m H_2SO_4, and 0.15 m $CuSO_4$ at 280°C. These rates are less than those observed in other 280°C loop experiments without appreciable amounts of solution additives. The specimen surfaces in L-2-19 were free of all but inappreciable amounts of sorbed uranium. In contrast, the specimens in the 0.04 m UO_2SO_4 experiment at 300°C, L-2-17, the only ones previously examined, showed appreciable amounts of surface uranium.

The probable importance of sorbed uranium to the radiation corrosion of Zircaloy-2 has been recognized and was discussed previously. The reduced rates in the L-2-19 experiment appear to be related to the absence of uranium in the corrosion-product film. This may be regarded as evidence that the

beneficial action of the solution additives is, at least in part, through an inhibition of the sorption of uranium. (AEC Activity 4103.1)

In-Pile Autoclave Slurry Corrosion Studies: LITR. — The effects of radiation on the corrosion of Zircaloy-2 exposed to slurry in which a deuterium overpressure was maintained were investigated in an autoclave test in the LITR HB-6 rocking-autoclave assembly. The autoclave was operated at 280°C for 1230 hr and was exposed to 770 hr of irradiation at an estimated average flux of 5×10^{12} neutrons·cm^{-2}·sec^{-1} and a power density of 0.6 kw/liter. The slurry contained 950 g of Th per kg of D_2O with 0.017 m MoO_3 as a recombination catalyst and was prepared from thoria which contained 0.5% of 93% enriched uranium. Corrosion rates, based on measurements of the increase in deuterium pressure, were 1.1 mpy for the first week and 0.4 mpy when averaged over the entire run. Analysis of the data taken during the run indicated that a rate of 0.3 mpy was attributable to radiation effects.

No measurable quantity of radiolytic gas was produced at 280°C. At 175°C, recombination rates of radiolytic gas equivalent to 0.055 mole of D_2 per liter-hour at 100 psi radiolytic D_2 (or 50 psi O_2) were observed, and a G_{D_2} of 0.02 (or G_{O_2} = 0.01) was obtained. The G values are lower by a factor of 20 to 30 than the values obtained for the previous oxygenated run at 280°C. (AEC Activity 4103.1)

Autoclave Radiation Corrosion Studies: LITR. — In two previous experiments, Z-13 and Z-22, operated as perpendicularly rocked autoclaves in facility HB-5 with depleted uranyl sulfate solutions (0.17 m UO_2SO_4 and 0.02 m $CuSO_4$ at 250°C), an anomalous pressure behavior was observed. No radiolytic-gas pressure was anticipated, but, in each case, the pressure within the autoclave started to increase following the initial exposure to radiation. The pressure reached a peak after about 60 hr of radiation and then decreased to essentially zero excess pressure. It has been postulated that this increase was due to a loss of copper catalysis by a loss of a large fraction of the copper ion from solution, possibly by plating out as metallic copper on the corroding metal, and to an inhibition of gamma-ray catalyzed combination of H_2 and O_2 by a small amount of copper remaining in solution. The copper, in this postulated picture, must re-enter the solution after being lost, in order to account for the return of the radiolytic-gas pressure to the normal negligible value. To test this general postulate, an experiment has been carried out with a depleted uranyl sulfate solution containing no copper, 0.17 m UO_2SO_4 only, at 250°C and in an axially rotated autoclave in the LITR facility HB-5. No increase in pressure or other unusual effects were detected. A similar experiment operated at 280°C exhibited a similar normal behavior. As mentioned, the former experiments were of the type in which the autoclave is rocked perpendicularly, and the latter were of the axially rotated type. The neutron distribution within these two types of experiments is also different. The former type of autoclave is exposed to an essentially constant neutron flux; the latter is exposed to a flux which decreases linearly by a little more than a factor of 2 from the forward to the rear end of the autoclave. The effect of these differences between the two types of experiments on the pressure behavior is unknown. Tentatively, the results are regarded as evidence in support of the above-mentioned postulate. (AEC Activity 4103.1)

CIVILIAN-POWER ADVANCED GAS-COOLED REACTOR (GCR)

Gas-Cooled Reactor: Design. — A study was initiated of the suitability of graphite-moderated gas-cooled reactors for breeding with the Th-U^{233} cycle. Preliminary results indicate that practical configurations exist which would permit initial conversion ratios of 1.1 or greater with U^{233} and that a net breeding gain appears possible, even with stainless steel fuel sheathing. An appraisal of the burnup characteristics of such reactors and of their economic potential is required.

Work continued on the problem of the surface temperature distribution in rod-cluster fuel elements. For fuel elements like those of GCR-2, a rod spacing can be found which will produce no temperature difference between the inner rod and the inward and outward facing surfaces of the outer rods of a seven-rod cluster. There will still be a temperature variation around the surfaces of the outer rods. The situation appears to be unstable, however, in that slight deviations of the capsule from its proper position may produce an unsymmetrical temperature distribution which will cause the deviation to increase still further. Solutions to this problem, such as decreasing the length of fuel elements, inserting spacers, or promoting greater turbulence in the coolant stream, will be investigated in an experimental system now being constructed.

Broad parametric studies of reactor—steam system performance as a function of the major parameters such as coolant temperatures and flow rates have confirmed the selection of parameters made in the GCR-2 study. For example, it is found that with a nominal maximum surface temperature of 1200°F, the most favorable coolant outlet temperature is about 1000°F. (AEC Activity 4119)

Gas-Cooled Reactor: Materials Compatibility Tests. — The program of tests of the compatibility of graphite and various metals of interest for fuel element cladding in a helium atmosphere was continued. Two tests in which the helium was circulated by thermal convection in a type 316 stainless steel loop were run for 1000 hr at a hot-leg temperature of 1400°F and a cold-leg temperature of 500°F. In the loop which contained TSF graphite, the attack of the metal specimens (various steels, zirconium, zirconium-base alloys, tantalum, and molybdenum) was similar to that found in static tests, ranging from severe scaling of zirconium and its alloys to barely detectable carburization of the stainless steels. Approximately 2 g of material was distributed in a definite pattern in the cold leg. Analyses showed the deposit to be 90% SiO_2 (alpha quartz) with 1 or 2% carbon.

In the loop which contained AGOT graphite, the specimens showed more reaction than those in the loop containing TSF graphite. Stainless steel samples had much greater weight gains, and low alloy steels showed gross attack. The T-1 steel sample, for example, had a very heavy, loosely adherent scale. A cold-leg deposit was present as in the test with TSF graphite. Analyses of the deposit are under way. (AEC Activity 4119)

Gas-Cooled Reactor: Metallurgy. — The problems associated with the use of beryllium as GCR fuel element cladding material are being investigated. Edge-fusion and plug-type end closures were designed, and several 1/2-in.-dia, 0.020- and 0.040-in.-wall tubes were sealed successfully with plugs by using the edge-fusion technique and conventional inert-gas-shielded arc welding equipment. Attempts to join beryllium sheet specimens with similar fusion welds made with automatic equipment in a dry box were unsuccessful. The high restraint present in these welds caused cracks in the adjoining base metal.

Evaluation studies are being conducted on the recently constructed eddy-current instrument and coil system for the nondestructive inspection of GCR fuel capsule tubing. Indications are that discontinuities 1 to 2 mils deep are detectable in 0.750-in.-dia, 0.020-in.-wall, type 304 stainless steel tubing. Correlations of this method with ultrasonic, radiographic, and destructive examinations are being studied.

Creep-buckling tests of fuel capsule tubing were continued. A test was devised in which a hot streak at a temperature of 1500°F is produced along the wall of a tube held at 1300°F in order to simulate more accurately the conditions under which creep buckling of fuel capsules might occur. The first specimen tested consisted of a type 304 stainless steel tube with a 0.020-in. wall and a 0.010-in. diametral clearance between the fuel slugs and the capsule wall. The specimen was tested for 150 hr at an external pressure of 300 psi; no measurable deformation occurred. A similar specimen with no internal slugs has survived over 800 hr at a uniform temperature of 1300°F without buckling. It appears that the material has considerable resistance to the buckling phenomenon at temperatures of 1300°F and below. (AEC Activity 4119)

GENERAL REACTOR RESEARCH

Basic Reactor Research. — The reactivity changes caused by inserting small samples of solutions of $U^{235}O_2F_2$, $U^{233}O_2(NO_3)_2$, and H_3BO_3 into the water-filled center of a critical annulus of $U^{235}O_2F_2$ aqueous solution were utilized to evaluate $\bar{\eta}^{233}\bar{\sigma}_a^{233}/\bar{\eta}^{235}\bar{\sigma}_a^{235}$. The experimental value for this parameter was found to be 0.983 ± 0.006. With the use of "world values" for $\bar{\eta}^{235}$, $\bar{\sigma}_a^{235}$, and $\bar{\sigma}_a^{233}$ (BNL-325), the calculated value of $\bar{\eta}^{233}$ was 2.39 ± 0.06. The neutron energy spectrum at the center of the cylinder was essentially thermal, since 98.8% of the fissions induced in 0.002-in.-thick U^{235} metal foils that were placed there were caused by neutrons whose energies were less than the cadmium cutoff energy, nominally 0.5 ev. The annular critical cylinder was 6 in. ID, 15 in. OD, and 19.8 in. high. (AEC Activity 4202)

Metallurgical Materials and Processing. — Diffusion of magnesium into the zirconium cladding on uranium cores is being investigated as a means of altering the chemical reactivity of the epsilon phase. The epsilon phase is stable at temperatures below 600°C and is a potential explosive hazard in nitric acid solutions. Zirconium-clad uranium cores are being heat-treated to develop the epsilon phase at the interface between the core and cladding

and then treated with magnesium prior to chemical dissolution tests in which ordinary epsilon-containing alloys usually yield a potentially explosive surface. (AEC Activity 4203)

Reactor Safety:

Fuel Element Catastrophe Studies. — An earlier examination of the nature of radioactive particulate material released by the slow melting of an APPR stainless steel fuel plate revealed that only cesium, whose oxide is unstable at high temperatures, was found on the filters. A further experiment was completed in which the rate of release of cesium was measured for periods up to 10 min. An approximate half time of 3 min for vaporization of cesium was observed. These samples all released the krypton in less than 30 sec. In experiments which were performed in a stream of air, the stainless steel pellets burned with incandescence, and the release of cesium was somewhat faster as well as more complete. (AEC Activity 4252)

Reactor Safety: Studies for Pool-Type Reactors. — The fabrication of the aluminum tank which will be used to study the effect of voids in the reflector of a swimming-pool reactor is more than 50% complete. The effect of voiding the entire side of a pool-type reactor is being calculated on the Oracle with various return-current boundary conditions. (AEC Activity 4252)

Thorex Process. — The uranium product from the Thorex pilot plant contains 20 to 60 ppm of U^{232} and a trace of Th^{228}; during storage the activity builds up to a high level. When the uranium product was deacidified and passed through an ion exchange column containing sufficient Dowex 50 to sorb 10% of the uranium, some of the Th^{228} was sorbed, but little if any of the Ra^{224}. Immediate counting of the effluent stream showed a gamma activity about 50% of that in the feed; after four days, the gamma activity was only about 30% of that in the feed. The feed solution contained 217 g of U per liter in 0.04 M HNO_3.

Infrared spectra showed that the complexing of thorium nitrate or uranium nitrate by tributyl phosphate without diluent results in the displacement of water associated with the tributyl phosphate molecule. Absorption bands at 1190 and 1520 cm^{-1} were tentatively identified as being due to covalent nitrate in the thorium nitrate complex, and bands at 1182 and 1537 cm^{-1} as being due to covalent nitrate in the uranium nitrate complex. The percentage transmission at 1287 cm^{-1}, identified tentatively as being due to the phosphoryl group, did not change with thorium concentration. The transmission at 1272 cm^{-1}, however, increased with thorium concentration, and another couplet at 1300 cm^{-1} decreased. In like manner the transmission at 1037 cm^{-1} (the C-O-P wave number) did not change, but the couplet at 995 cm^{-1} increased, and the couplet at 1060 cm^{-1} decreased. (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Darex Process. — In the Darex process, fuel elements containing uranium and stainless steel are dissolved in dilute aqua regia, the chloride is distilled off, and the uranium is recovered by solvent extraction. Two engineering-scale runs of 5 and 20 hr duration were made in the Darex loop equipment. In the first run, 22 ft of prototype Yankee Atomic fuel was dissolved; in the second run, 75 ft was dissolved. Losses were approximately 1.0 mole of HNO_3 and 0.2 mole of HCl per mole of metal

dissolved. The stripped product in both runs contained less than 7 ppm chloride. In the short run, only the dissolver and stripper were operated; in the longer run, the feed adjustment tank was also operated satisfactorily with automatic control of its power input by control of the temperature.

Two continuous feed adjustment runs of 6.25 and 7.25 hr duration were made at 132°C with feed containing 87.5 g of U per liter, 19.1 g of stainless steel per liter, 13.4 M HNO_3, and 23 ppm chloride. The concentrated products before dilution with water contained 550 g of U per liter, 120 g of stainless steel per liter, 4.3 M HNO_3, and 30 ppm chloride; the overheads were 15.0 M HNO_3. All material balances were between 98 and 106%, out/in. In the temperature range 130 to 135°C, metal loading and H^+ in the product varied, although the total was constant; H^+ and nitrate in the overhead remained nearly constant. (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Materials Evaluation. — Titanium continues to behave well in corrosion tests simulating conditions in the Darex continuous dissolver and feed adjustment tank. In the Sulfex-Thorex system, scouting experiments on Nionel are promising. Carpenter-20Cb showed somewhat better resistance to cycling tests than was expected from the results of control tests in the two solutions. The Carpenter-20Cb specimens showed transcrystalline corrosion after exposure to sulfuric acid solutions.

Amercoat-74 epoxy coating exposed to 5×10^9 r of gamma rays in water underwent a color change but no apparent physical damage except a slight increase in thickness. The compressive strength of Haveg Nos. 41 and 61 increased 8.6% and 2.0%, respectively, after receiving 10^{10} r of gamma rays in water, and Haveg No. 093 decreased 7%. (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Zircex Process. — In the Zircex process, uranium-zirconium fuel elements are hydrochlorinated to form volatile $ZrCl_4$, the residue being processed for uranium recovery. In a 14-hr run, 98.7% of a 4.36-kg section of an STR subassembly reacted with anhydrous HCl that had been passed through an activated charcoal bed; the HCl utilization efficiency was 75%. The uranium present in material insoluble in 10 M HNO_3 was 3.4%, a twofold increase over that in the last run; the sublimate loss was 0.012%, the lowest yet achieved with STR fuel. In a second run, 54.3% of a 4.21-kg charge reacted in 5.5 hr; the run was shut down because of boiling in the caustic-filled absorbers for excess HCl. (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Alternate Aqueous Dissolution. — The tin in a Zircaloy-2 cladding was nearly completely dissolved by 6 M NH_4F to which 1.0 M NH_4NO_3 was added; the amount of hydrogen formed was decreased by the nitrate. Cupric chloride added to the 6 M NH_4F decladding solution also resulted in dissolution of the tin, but uranium losses were ~0.13%, as compared with ~0.02% with NH_4NO_3.

The rate of dissolution of Consolidated Edison sintered ThO_2-UO_2 pellets in 13 M HNO_3—0.04 M NaF was unaffected by the addition of $Al(NO_3)_3$ in concentrations up to 0.2 M. Approximately 98% of the thorium was dissolved in

7 hr in these solutions. Only about 92% of the thorium was dissolved in a solution containing 0.4 M $Al(NO_3)_3$.

A tentative flowsheet was developed for the dissolution of the EBWR fuel alloy (93.5% U—5% Zr—1.5% Nb) in 8 M HNO_3—1.0 M HF. The HF was added incrementally to control the rate of reaction. During the dissolution a precipitate formed which contained a high percentage of niobium and which was soluble in hydrofluoric acid. The precipitate contained 0.3% of the uranium. An alternative dissolvent, aqueous NH_4F, is also being evaluated, but at present it appears less attractive than HNO_3-HF in that the uranium is almost quantitatively precipitated during the dissolution. (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Solvent Extraction Studies. — Small engineering-scale tests were made with the TBP-Decalin system to obtain additional flooding data, which should permit the correlation of flooding rates with the physical properties of the aqueous and organic streams. Such a correlation should make possible the prediction of flooding conditions for any proposed power reactor flowsheet. For the system water and 35% TBP-Decalin (aqueous/organic volume ratio = 0.4), the log of the flooding rate decreased linearly as the pulse pumping rate increased over the range 50 to 100 cpm. (AEC Activity 4301)

Hermex Process. — Tentative solubility curves for uranium in mercury and in 0.1 wt % magnesium amalgam were determined. The solubility of uranium in mercury increased from 0.0048 to 1.1 wt % as the temperature increased from 25 to 356°C. The solubility of uranium in 0.1 wt % magnesium amalgam increased from 0.011 to 1.41 wt % as the temperature increased from 75 to 356°C. The solubility of ruthenium in mercury increased from 0.86×10^{-7} to 3.5×10^{-7} wt % as the temperature increased from 25 to 356°C. The separation factor for uranium from ruthenium in mercury may be as high as 2.3×10^7 at 356°C if a small-pored filter is used and if the presence of uranium does not affect the solubility of the ruthenium. (AEC Activity 4303)

Fused Salt—Fluoride Volatility Process. — In a 2-hr fluorination at 600°C of 50-50 mole % $NaF-ZrF_4$ containing 14 wt % uranium as UF_4, the uranium loss was only 0.014%. This is nearly twice the uranium concentration, 8 wt %, used in previous work.

A test dissolution of Zircaloy-2 by HF, with NaF-LiF as the fused salt mixture, was carried to completion at an average rate of 5.56 $mg \cdot min^{-1} \cdot cm^{-2}$ (0.683 $lb \cdot hr^{-1} \cdot ft^{-2}$) with an HF feed rate of 2.0 lb/hr. The reaction proceeded smoothly except for an unexplained difficulty in transferring the salt melt at the end of the test. (AEC Activity 4302)

Waste Treatment and Disposal. — In studies on waste calcination, to obtain data for design of a waste-processing pilot plant, the amount of fission products volatilized during heating of synthetic wastes was determined as a function of temperature. The amount of activity volatilized increased rapidly as the temperature was increased above 150°C, the beta leveling off at

400°C and the gamma at 700°C. The equilibrium amounts in the off-gas were 1.0% of the original beta and 4.5% of the gamma. The results of leaching the calcined solids with water were erratic, but cesium was consistently the most abundant element in the lixiviums.

In calcination tests on simulated stainless-steel-containing wastes, constant weight was reached at 500°C; with wastes containing zirconium fluoride, aluminum nitrate, nitric acid, or stainless steel plus MgO, constant weight was not reached below 700°C. The freezing points of all five synthetic wastes were below 14°F. Neutralization of nitric acid wastes with NaOH or CaO, followed by filtration, resulted in a cake with a volume half that of the original waste. The volume of the cake from a similar treatment of stainless-steel-containing waste was about one-fifth that of the original. The neutralized wastes froze at ~32°F.

In studies on the disposal of the Maritime Reactor waste, trace fission products were sorbed on a mixed-resin bed, the filtered resin was embedded in portland cement, and, after hardening, the cement block was placed in synthetic sea water. After 17 days, 7.7% of the gamma and 2.9% of the beta activity had been leached. (AEC Activity 4305)

PROGRAM 5000 — PHYSICAL RESEARCH

Reactor Operations. — Tests on heating the Graphite Reactor with air blown through tubes inserted in each channel indicated that the insulation on the tubes will have to be improved. The graphite appeared to be heating satisfactorily, although the test had to be discontinued to prevent overheating of the concrete.

ORR Project. — The testing program on the ORR is continuing. Tests have been completed on measurements of control-rod worth and the effect of various experimental facilities and dummy experiments. Measurements have been made on the thermal-neutron flux distribution in typical core loadings with and without dummy experiments in position. At the present time, various tests are being conducted at power levels up to 10 Mw.

PHYSICS

Neutron Velocity Selector. — Total-cross-section measurements have been continued with the M-1 chopper at the LITR. The nuclides which have been measured recently include Pm^{147}, I^{129}, Zr^{93}, Sm^{151}, Sm^{149}, Sm^{147}, Eu^{151}, and Eu^{153}. Resonance parameters have been determined for the fission products Pm^{147}, I^{129}, and Sm^{151}; the values of the strength functions ($\bar{\Gamma}^{o}_{n}/D$) and

average level spacings per spin state derived from the resonance parameters are $(4.2 \pm 1.2) \times 10^{-4}$, (8 ± 2) ev; $(0.4 \pm 0.2) \times 10^{-4}$, (60 ± 20) ev; and $(2.0 \pm 0.9) \times 10^{-4}$, (2 ± 0.6) ev, respectively. (AEC Activity 5220)

High Voltage Program. — The techniques of the pulsed Van de Graaff and of millimicrosecond time-of-flight have been applied to the difficult problem of neutron capture in the kev energy range. A large (~250 gal) tank of liquid scintillator has been constructed to serve as the gamma-ray detector. The tank has a 12-deg truncated conical opening through the center. Neutron bursts travel through this hole and interact with a thin sample placed at the geometrical center of the tank.

The time resolution of the tank and associated electronics is about 10 mμsec for a single gamma ray of 3 Mev or more energy. Capture cross sections relative to capture in silver have been measured for neutrons near 30 kev for the elements given in the table below. These measurements were performed with "kinematically" collimated neutrons, that is, by using neutrons produced at forward angles only in the $Li^7(p,n)$ reaction. A more general but more difficult approach is straightforward collimation by shielding, which permits measurements of capture as a function of energy. Preliminary tests with a massive neutron collimator indicate the feasibility of measuring capture as a function of energy in the kev range, at least for elements where $\sigma_a \gtrsim 100$ mb.

Neutron Capture Cross Sections at 29 ± 8* kev

Sample	Z	σ** (mb)	Sample	Z	σ** (mb)
C	6	≤ 0.5	Cd	48	367
Fe	26	15	Sn	50	102
Ni	28	40	Ta	73	660
Cu	29	59	Pt	78	350
Mo	42	182	Au	79	455
Ag	47	(920)	Pb	82	19

* Full width at half maximum; full width at base is ±14 kev.
** Based on σ = 920 ± 90 mb for Ag. Errors for other samples (except C) estimated as ±15% or ±10 mb.

The (p,n) cross sections for 14 isotopes of the elements Pd, Cd, In, and Te at proton energies between 3 and 5.5 Mev have been compared with the compound nucleus cross sections that are predicted by a totally absorbing square well of radius $1.45A^{1/3} \times 10^{-13}$ cm. Since the (p,n) cross section is essentially that for formation of the compound nucleus, the ratio of the observed to the predicted cross section is equal to the ratio of the observed to the predicted proton strength function averaged over all partial waves. The observed over-all average ratio is about 1.5; however, there are systematic deviations from the average. A general rise of about 15% occurs from A = 105 to A = 125. For a given element the strength function increases slightly with mass number, about 15% from Cd^{111} to Cd^{116}. For the isotopes of Pd and Te there is no significant energy dependence; however, for Cd and In there is a definite rise in the strength function with increasing proton energy. (AEC Activity 5220)

Charge Spectrometry. — The relative abundance of the various charged fragments formed following the beta decay of monotritiated benzene has been measured. Thirty-one singly charged fragments were found. Of the total ion current, only 0.1% is represented by $(He^3)^+$, while the bulk, about 70%, is found as $C_6H_5^+$. The C-He bond probably breaks in most cases after a single vibration, the $C_6H_5^+$ being often left with enough energy to dissociate into further fragments. The presence of about 3% of the total number of fragments in the form of a carbon skeleton completely stripped of hydrogen (C_n, where $n = 1$ to 6) suggests the existence of a fairly energetic process, since from electron impact studies such fragments have appearance potentials from 40 to 45 ev. Further, since the calculated maximum energy available from the recoil of beta emission in C_6H_5T is only 3 ev, some extra excitation from the nonadiabatic nature of the process must be postulated. In general, the spectrum of the tritiated benzene is more spread out than in the electron impact study, reflecting the probability of a greater spread of excitation than that represented by the energizing by monoenergetic electrons. (AEC Activity 5220)

Neutron Diffraction. — A neutron diffraction study has been made of the magnetic properties of the rare-earth iron perovskites $NdFeO_3$, $HoFeO_3$, and $ErFeO_3$, at temperatures ranging from 955 to 1.25°K. The iron ions in each of these compounds undergo a transition to an antiferromagnetic configuration in which each moment has six oppositely directed moments at nearest-neighbor distances. The Néel temperatures are 760, 700, and 620°K, respectively, for the compounds of Nd, Ho, and Er. The moment directions in $HoFeO_3$ and $ErFeO_3$ are parallel and antiparallel to the orthorhombic [100] direction at room temperature; at 43°K the moments are found to be in a $(1\bar{1}0)$ plane. In $HoFeO_3$ the iron ion moments at 1.25°K are parallel to [001]; in $ErFeO_3$ at the same temperature they are parallel to [110]. The magnitudes of the ordered iron moments at temperature saturation are 4.5_7, 4.6_0, and 4.6_2 Bohr magnetons in $NdFeO_3$, $HoFeO_3$, and $ErFeO_3$, respectively. In the liquid-helium temperature range, magnetic ordering transitions of the rare-earth ions in $HoFeO_3$ ($T_N = 6.5$°K) and $ErFeO_3$ ($T_N = 4.3$°K) are observed. The Er^{3+} ion moments form a nearly ideal antiferromagnetic configuration in which a chain of parallel moments is surrounded by four chains of oppositely directed moments at nearest-neighbor distances. In this compound the Er^{3+} ion moments are parallel and antiparallel to [001] and have at 1.25°K a magnitude of 5.8 Bohr magnetons. In $HoFeO_3$ the ions are ordered in a distorted antiferromagnetic configuration in which, at 1.25°K, each Ho^{3+} moment with a magnitude of 7.5 Bohr magnetons makes an angle, in the (001) plane, of about 27 deg with the [010] direction so as to produce a net ferromagnetic moment of 3.4 Bohr magnetons per $HoFeO_3$ molecule parallel to [100]. (AEC Activity 5220)

Thermal Diffusion. — Krypton-86 in the amount of 100 cc at approximately 0.9 atm pressure was removed from the thermal diffusion columns. The assay was 99.6% Kr^{86}, with the remainder Kr^{84}. Now that the gradient has been established after some ten months of operation, it should be possible to produce further amounts of the heavy isotope at a rate limited only by the rate of transport in the columns. (AEC Activity 5250)

CHEMISTRY

Raw Materials Chemistry. — In continuous runs demonstrating the use of a two-cycle Amex process for recovering uranium and thorium from monazite sulfate liquors, >99% of the uranium was recovered in the first extraction cycle with a tertiary amine (triisooctyl). The uranium was stripped with sodium carbonate solution. In the thorium recovery cycle, the amount of phosphate extracted with the thorium depended strongly on the choice of amine extractant. Large amounts of phosphate were extracted with a secondary amine (ditridecyl), whereas very little phosphate accompanied the thorium in extractions with a primary amine (Primene JM-T).

Two new N-benzyl-(secondary alkyl)-amines had uranium extraction coefficients nearly as high as did the N-benzyl-(branched secondary alkyl)-amines previously reported (ORNL-1886, -2506). These are N-benzyl-1-n-nonyl-n-decylamine and N-benzyl-1-n-undecyl-n-dodecylamine. Solutions in kerosene and in benzene, 0.1 M, gave extraction coefficients of ~2000 from 1 M sulfate solution at pH 1, as compared with coefficients in the range 20 to 200 with other typical amines. These amines have a better prospect of becoming commercially available than do the more highly branched N-benzyl amines previously tested.

A sorbant prepared by precipitating zirconium with phytic acid (inositol hexaphosphoric acid) removed 97% of the cesium (initially 10 ppm) from 1 M aluminum nitrate solution in a single contact at 1 g per 100 ml, and 80% from 0.5 M sodium nitrate solution. The equilibrium pH's were 1.6 to 1.8. The separation factor from sodium in the sodium nitrate solution was estimated to be of the order of 100. (AEC Activity 5310)

Americium Recovery. — The first half of the americium product recovered from Los Alamos concentrate in the Metal Recovery Plant was separated from impurities by factors of 30 to 3000 by citrate elution from cation exchange resin followed by fluoride and hydroxide precipitations. The impurities, in grams, were as follows:

	Original	Final		Original	Final
Pu	0.7	0.015	Cr	140	0.04
La	30	0.95	Ni	88	0.06
Fe	790	0.57	Al	500	0.45

The product contained 17.1 g of americium, which represents 90% recovery; the other 10% will be recycled. (AEC Activity 5310)

Neptunium Recovery. — In laboratory countercurrent batch extractions, a modified Neptex flowsheet gave a separation factor of neptunium from thorium of about 10. The feed was adjusted to 50 g of U per liter, 1.8 M $Al(NO_3)_3$, and 3 M HNO_3 containing neptunium and thorium. The extractant was 15% tributyl phosphate, the scrub was 1.5 M $Al(NO_3)_3$—3 M HNO_3, and the feed:extractant:scrub flow ratio was 1:1.2:0.33. In an extended run with 12 volume changes, 6.6% of the thorium was extracted, and 3.1% of the neptunium remained in the aqueous phase. (AEC Activity 5310)

Extraction Reagent Performance. — Irradiations with nominal 1-Mev electrons at dose densities of the order of 1000 to several thousand times those encountered in Thorex process first-column conditions were performed on four tributyl phosphate—Amsco systems containing dissolved nitric acid. The G values for gas, at dose densities of 420 to 480 whr/liter, were less than the corresponding values for either pure TBP or pure Amsco. Emulsification tendencies of systems using TBP may be largely due to a polymer formed during radiolytic decomposition of TBP, rather than to the decomposition product dibutyl phosphoric acid, at irradiation levels of 25 to 100 whr/liter.

Radiation decomposition products increased the extraction coefficient of diethyl carbonate, but nitric acid decreased it. Both radiation decomposition products and nitric acid decreased the stripping coefficient, S_o^a. (AEC Activity 5310)

Feed Materials Processing. — Varying the temperature, the sodium excess, and the methods of contacting failed to increase the metal reduction yield of uranium from UF_6 by sodium amalgam to above 40%.

Approximately 85% of the uranium in UF_6 contacted with 0.8 M lithium amalgam in a high-shear turbomixer at 25°C was reduced to uranium metal. An excess of lithium and temperatures below 100°C are favorable for the reaction.

In studies on the Druhm process, the direct reduction of uranium hexafluoride to metal by sodium, four uranium hexafluoride reduction runs were made in order to determine the requirements for the UF_6 entrance nozzle. A uranium material balance on the first run was 105.6%. The maximum theoretical flame temperature for the Druhm reaction was calculated to be 1977°K. (AEC Activity 5310)

Ion Exchange Technology, Chemical Technology Division. — In preliminary differential-bed experiments on rates of exchange of uranium between uranyl sulfate solutions and Dowex 21K anion exchange resin, initial rates were very high: 50 to 100 mg of U per minute per gram of dry resin. As the resin loading increased to 50%, the rates decreased rapidly, to 5 to 10 mg of U per minute per gram of dry resin, and decreased slowly thereafter.

As part of the program to optimize the conditions for recovery of uranium from sulfate solution by ion exchange, the concentrations of the various ionic species in the UO_2SO_4-H_2SO_4-Na_2SO_4-H_2O system were calculated on the IBM-704. The calculations will be extended to systems containing resin. (AEC Activity 5310)

High-Level Nuclear Chemistry. — The most troublesome separation encountered in the isolation of large quantities of neptunium from diffusion process ash arises from the high ratio of thorium to neptunium in the recovery-plant product. An especially promising method involving the use of 5% tributyl phosphate was investigated. Catalytic oxidation of neptunium to Np(VI) in 6 M HNO_3 was first accomplished by the addition of sodium nitrite. Under

these conditions, distribution coefficients greater than 2.8 for neptunium have been obtained, while those for thorium remain at about 0.1. (AEC Activity 5330)

Chemistry of Corrosion. — The reduction of uranyl ion on the surface of passive electropolished stainless steel in an oxygenated solution of 0.010 f H_2SO_4 + 0.090 f Na_2SO_4 at 85°C was studied potentiostatically. In agreement with earlier studies at 44 and at 67°C, the reduction reaction is first order with respect to the concentration of uranyl ions in solution over the whole range of potentials and concentrations examined, and each reduction act involves the transfer of two unit charges across the electrode-solution interphase. The differential capacity of the passive stainless steel interphase was measured as a function of electrode potential in the chosen environment, and it showed no change as a function of uranyl ion concentration.

Electrochemical measurements were completed for the zirconium-oxygen corrosion system in oxygenated acid sulfate solutions at temperatures from 25 to 90°C. Corrosion rates and other kinetic constants were calculated from the data. By using a model which postulates a decreasing electric field in the corrosion film as the film thickens, a rate law was derived which fits the experimental rate-time behavior closely. A convenient and exact analytical expression cannot be given, but the rate law is roughly a hyperbolic law of the form, rate = $K/(t + t_o)$, where K and t_o are constants. (AEC Activity 5330)

Ion Exchange Studies. — Ionogenic compounds soluble in organic solvents but insoluble in water have recently become available. Solutions of these compounds in organic media can selectively extract ions from aqueous solutions and thus serve as liquid analogues to the more commonly used resinous exchangers. Dimethyldidodecenylammonium chloride, a quaternary amine salt soluble in toluene, was used to study the extraction of Zn^{65}(II) from HCl and LiCl solutions. The dependence of the distribution coefficient on electrolyte concentration was very similar to that observed with Dowex 1 and other anion exchangers. Selectivity coefficients for the extraction as a function of loading were also measured for the bromide-chloride system with varying concentrations of quaternary ammonium chloride dissolved in toluene. It was found, as in previous experiments with variable-capacity resinous exchangers, that the selectivity coefficient increases as "capacity" (i.e., concentration) in the exchanger phase decreases.

Measurements of the heat of exchange in the iodide-bromide system were made by using Dowex 2 resins of nominal 0.5, 2, and 24% divinylbenzene (DVB) cross-linking. The heat of exchange with the 24% DVB preparation was -1650 cal/mole, with little change as the resin composition varied. With the 0.5% DVB resin the differential heat varied from -2600 cal at $x_{Br} = 1.0$ to -1000 cal at $x_I = 1.0$. The shape of the differential heat—composition curve followed approximately the water content (x_w) curve for the same resin. With cation exchangers a fair approximation to the heat of exchange can be given by the differences in heats of dilution of several salts. A similar calculation for anion exchangers with H^+, Li^+, and Na^+ as the cations showed no discernable correlation. (AEC Activity 5330)

Chemistry of Technetium. — Further investigations of the influence of molecular structure on the solvent extraction of pertechnetate ion from aqueous solutions by use of 1-, 2-, and 3-pentanol and 2-, 3-, and 4-heptanone showed that as the five-carbon-atom alcohol molecule became more symmetrical, the extraction improved, whereas extraction decreased with ketones when the carbonyl group came nearer to the center of the molecule. Tertiary alcohols, however, were the most efficient extractants of all the straight or branched-chain aliphatic alcohols. Measurements with Tc(V) and Tc(IV) showed these valence states to be extracted by ethers when excess thiocyanate or chloride ion was present, in contrast with Tc(VII), which was not extracted.

The compounds K_2TcBr_6, K_2TcI_6, and K_2ReI_6 were prepared, and their visible and ultraviolet spectra in various solvents were observed. Potassium hexabromotechnetate (red crystals) dissolved in distilled concentrated HBr gives a red solution with an absorption spectrum exhibiting absorption bands at the following wavelengths: 7450, 4450, 3840, 3260, and 2600 Å, with molar absorbancy indices of 23, 5900, 6940, 12,300, and 18,900, respectively. Solution of the compound in 2 M H_2SO_4 to permit observation of the spectra at lower wavelengths reveals an intense band at 2175 Å.

A solution of K_2TcI_6 in 6 M HI is not stable; a 3×10^{-4} M solution (deep violet-red) becomes essentially colorless overnight. Similar to the corresponding rhenium compound, the crystals of K_2TcI_6 are lustrous black.

The absorption spectrum of a 6 M HI solution of K_2ReI_6 shows nine absorption bands between 3500 and 8000 Å, with maxima at the following wavelengths (molar absorbancy indices given in parentheses): 6980 (1930), 6640 (2380), 5750 (5280), 5370 (6320), 5220 (7020), 4830 (4810), 4400 (7210), 3880 (8540), and 3525 Å (13,200). The 4400-Å band is resolved into three bands with maxima at 4475, 4325, and 4200 Å when the K_2ReI_6 is dissolved in 2 M H_2SO_4 or in dilute HI. The 2 M H_2SO_4 solution is not stable at room temperature; the solution becomes essentially colorless in 30 min. Cooling the solution to ice temperatures increases its stability. (AEC Activity 5330)

Characterization of Short-Lived Fission Products. — Extensions of both the single-crystal scintillation counter and the coincidence rate measurements previously reported on 2.9-min Rb^{90} were made with the use of a Radiation Counter Lab. 256-channel pulse-height analyzer. The spectrum obtained with a single 3 x 3 in. NaI crystal spectrometer of improved resolution (7.6% at 0.662 Mev) was analyzed into gamma-ray components of 0.52, 0.72, 0.833, 1.00, 1.10, 1.23, 1.39, 1.68, 1.81, 2.15, 2.51, 2.73, 2.85, 3.05, 3.34, 4.12, 4.34, 4.60, 5.08, and 5.23 Mev. In coincidence with the intense 0.84-Mev gamma-ray transition were gamma rays at 0.52, 0.72, 0.87, 1.05, 1.11, 1.24, 1.41, 1.71, 1.82, 2.20, 2.51, 2.70, 2.83, 3.35, 3.56, and 4.4 Mev. Gamma rays at 0.5, 0.836, and 1.06 Mev appeared in coincidence with the 3.34-Mev gamma ray.

The most energetic beta ray had an end point at 6.59 ± 0.15 Mev. Beta rays at 5.81 ± 0.10 and 4.4 Mev appeared in coincidence with the 0.84-Mev gamma ray. With the window of the pulse-height selector set on the 5.23-Mev

gamma-ray photopeak, a coincident beta group at 1.2 Mev was observed; when the window was set to trigger on all gamma rays greater than 3.8 Mev, the beta ray in coincidence had an end-point energy of 2.21 ± 0.08 Mev.

Measurements with an end-window proportional counter and with a 4π beta counter gave a half life for Rb^{90} of 2.90 ± 0.09 min. The number of 0.84-Mev gamma rays per beta disintegration was found to be 0.61.

It has been possible to formulate a partial decay scheme from the above data. (AEC Activity 5330)

High-Temperature Aqueous Solution Chemistry. — Some preliminary phase equilibrium data for the five-component system UO_3-NiO-CuO-SO_3-D_2O were obtained at 300°C in the concentration ranges 0.05 to 0.10 m SO_3. Although of a fundamental nature, these investigations are related to fuel stability questions for the HRT operation.

A differential manometer apparatus was constructed to measure accurately small vapor-pressure lowerings in aqueous systems at high temperature. The vapor-pressure lowering of a 0.25 m Na_2SO_4 solution has been measured from 175 to 320°C in preliminary tests of the apparatus. This program is directed toward the measurement of $\Delta P/P_{solvent}$ values for UO_2SO_4-H_2O solutions and aqueous systems in general. (AEC Activity 5330)

Chemical Separation of Isotopes. — Separation factors were determined for lithium isotopes between aqueous lithium ions and lithium ions on Dowex 50 resin. The influence of the degree of cross-linking on the selectivity of lithium isotopes was investigated in a series of experiments using Dowex 50-4x, -12x, and -24x. A band elution technique was used. The number of theoretical plates in the columns was determined from the elution curve by the method of Glueckauf. The total isotopic separation (S) was calculated from the isotopic analysis of the front and rear edges of the eluted lithium band. The experimental data are presented in the following table:

Cross-Linkage	Mesh Size	Column Size (mm)	Eluant HCl (N)	Band Travel (cm/sec)	Plates (theo)	S	α
4x	200—400	15 x 650	0.1	2.3×10^{-3}	1000	1.29	1.0003
4x	200—400	15 x 650	0.1	1.3×10^{-3}	333	1.12	1.0003
12x	200—400	19 x 1120	0.23	5.5×10^{-4}	106	1.43	1.003
24x	100—200	15 x 650	0.3	3.4×10^{-3}	138	1.50	1.003

A tenfold increase in the enrichment factor, $\alpha - 1$, was observed as the cross-linking of the resin was increased from 4 to 12%. No further increase was noted on going to 24% cross-linkage. The increase in separation factor was probably due to the stripping of water of hydration from the lithium ions in the resins of higher cross-linking. (AEC Activity 5340)

METALLURGY

Radiation Effects on Oxidation. — An investigation has been undertaken to determine the effect of reactor radiation on the gaseous oxidation of metals. An apparatus for the in-pile oxidation of niobium has been built and is in the process of being tested. Previously obtained out-of-pile oxidation data for niobium (ORNL-2422) will be used as a basis for evaluating radiation effects in these new experiments.

A study of the oxidation of copper is also in progress with the eventual aim of determining the effect of radiation on the oxidation properties of this metal. Since the oxidation mechanism for copper is known to be different from that for niobium, a comparison of the results of these two studies should further assist in an assessment of the effect of radiation on oxidation. Electron-optical studies of the oxide films on copper single crystals have been undertaken to characterize the oxide films formed on copper in the absence of radiation fields. The temperature range of these experiments has extended from 70 to 300°C. It was found that at the lower temperatures the oxide on the (311), (110), and (111) crystal planes of copper developed marked thickness inhomogeneities, the underside of the films being rougher than the surface. The oxide on the (100) crystal planes was much more uniform. The epitaxial relationships between the oxide and substrate were also investigated. All these data will be used as a basis for assessing changes in the oxidation characteristics of copper in the presence of radiation fields. (AEC Activity 5420)

High-Temperature Reactions of Metals and Ceramics. — An electron-optical study of oxide films formed on several metals in groups IV and V of the periodic table was undertaken to determine the cause of nonprotective oxide formation on these metals. Small, blister-like cracks, similar to those previously reported for niobium oxide, were observed in oxide films on tantalum, and the formation of these cracks could be qualitatively correlated with an acceleration of the oxidation rate of tantalum.

Similar investigations have been carried out with vanadium, titanium, and zirconium, but no blister formation was observed in the oxide films formed on these metals. At relatively high temperatures, oxide films on both titanium and zirconium developed highly faceted surfaces, while in the temperature range 400 to 600°C, titanium oxide exhibited pronounced whisker growth. Zirconium oxide films showed none of these features but appeared to reflect the surface topography of the original metal surface. (AEC Activity 5420)

BIOLOGY

Cytology and Genetics: Plants and Fungi. — Experiments on the interaction between the mutagenic effects of chemicals and x rays have been continued. It was found earlier that x rays decreased the frequency of the reverse mutations induced by chloroethyl methanesulfonate. Recent experiments make it likely that this decrease is due to a differential killing by x rays of the chemically induced mutations, rather than to a mutation-preventing mechanism. Experiments are in progress to check this point with microconidia. (AEC Activity 6130)

Cytology and Genetics: Insects. — Earlier studies on lily chromosomes (Science 119, 45, 1954) showed that all x-ray aberrations induced at the first meiotic metaphase involve half-chromatid breaks and exchanges. The scoring of the x-ray effects was done at the first meiotic anaphase, 2 hr after irradiation. Study of the second meiotic anaphase following irradiation of the first metaphase has now been undertaken. Four doses of x ray were used: 5, 10, 15, and 20 r. Preliminary counts show that (1) the half-chromatid exchanges persist until the second anaphase (16 hr at 22°C), (2) the frequency of half-chromatid bridges at the second anaphase is linear with dose and approximately equal to the frequency of first-division bridges, and (3) the chromatids at second anaphase have nonstaining "gaps" along their length; the frequency of such gaps with dose has yet to be determined. This will be an important determination because the section of chromatid distal to such a "gap" may represent a chromatid fragment produced by the breakage at first metaphase of two half chromatids within the same chromatid. (AEC Activity 6130)

Pathology and Physiology. — Male mice of the RF strain were exposed to 450 r of whole-body 250-kv-peak x radiation at 10 weeks of age. Immediately after irradiation, groups of 70 to 90 animals each were injected intravenously with sterile Tyrode's solution, with bone marrow cells from nonleukemic mice of the C3H or AK strains, or with cell-free filtrates of brain tissue or of pooled brain, spleen, thymus, and lymph node tissue from AK mice bearing transplanted lymphomas. The incidence of thymic lymphomas in the various groups was as follows: nonirradiated controls, 1%; 450 r plus Tyrode's solution, 5%; 450 r plus AK bone marrow, 15%; 450 r plus C3H bone marrow, 14%; 450 r plus filtrate of pooled AK leukemic tissues, 12%; 450 r plus filtrate of AK leukemic brain, 23%. A similar lymphoma-inducing action of AK tissues or tissue filtrates, noted earlier in neonatal RF recipients (Proc. Soc. Exptl. Biol. Med. 93, 165, 1956), has not been observed in nonirradiated adult recipients of the RF strain. These results indicate that x irradiation may render the adult susceptible, like the newborn, to the leukemogenic action of subcellular agents present in tissues of mice of a high-leukemia strain. (AEC Activity 6130)

Cell Physiology. — Work continued on the isolation and characterization of peptides and other ninhydrin-positive compounds from rat liver microsomes. In the course of this work, spermine, a polyamino compound, was identified. This compound, isolable in significant quantities from liver microsomes, is toxic when injected into rats, producing vertigo and respiratory failure.

It was also shown that peptides can be isolated from unfertilized frog eggs and developing embryos. This finding will be the basis for future work aimed at correlating changes in peptides with stages in embryonic differentiation.

The antigenic components of rat liver pulps have been further examined. Agar diffusion studies indicate that there are several antigens which are common to nuclei, mitochondria, microsomes, and soluble phase and blood serum, in addition to a few fraction-specific antigens. The results suggest that there are determinant groups in mitochondria which do not react visibly with antimitochondria sera, whereas homologous determinant groups in other fractions are so organized as to give a visible reaction with antimitochondrial sera. (AEC Activity 6130)

Mammalian Recovery. — Protection of mouse bone marrow cells x-irradiated (800 r) in vitro has been obtained by removing most of the oxygen from the cell suspension just prior to exposure. Protection is judged by the ability of the irradiated marrow cells to promote recovery in mice exposed to a lethal dose of x rays.

Red blood cell repopulation in irradiated mice is markedly depressed if an artificial polycythemia is produced in the mice that serve as donors of the bone marrow. The repeated blood transfusions given to the donor mice depress their red blood cell forming tissue.

Oral administration of aspirin, atabrine, nilevar, reserpine, terramycin, or terramycin plus reserpine did not reduce the foreign bone marrow reaction in irradiated mice given rat bone marrow. Diets deficient in specific vitamins were also not effective.

Tissue changes in the spleens of irradiated mice given foreign bone marrow were identified and probably represent the earliest detectable evidence of the foreign bone marrow reaction. These changes were present at 24 and 48 hr after irradiation and after injection of foreign bone marrow. (AEC Activity 6230)

Radiation Immunology. — Serum protein studies in mice of various age groups showed that the lowest ratio of albumin to globulin (0.4) occurred in 1-week-old mice, followed by a marked rise through the 4-week-old group, and then a slight drop to ~0.8 in mice from 6 to 30 weeks of age. The albumin-to-globulin ratio of males and females did not differ in the 1- to 12-week-old mice; however, among the 20- to 30-week-old mice the serum of the females showed a significantly higher ratio of albumin to globulin.

A study of the antibody response of mice to a closely related heterologous red blood cell antigen during the maximum depression period after varying doses of x irradiation showed a curvilinear relation for the radiation dose and antibody response. The indices for antibody response were induction period, rate of appearance of circulating antibodies, mean peak titer, and mean total titer.

The results are compatible with the hypothesis that the radiosensitive recognition mechanism of antibody-producing cells of normal adult mice can differentiate closely and distantly related antigens with equal efficiency, but after irradiation the recognition mechanism for the more closely related antigen is damaged more than that for a distantly related antigen. (AEC Activity 6230)

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