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STATUS AND PROGRESS REPORT

JUNE 1958

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

June 1958

Date Issued: JUL 1958

OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee
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UNION CARBIDE CORPORATION
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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

June 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. — The processing of Paducah fluorinator ash for the recovery of Np^{237} and uranium was continued. To date, a total of 13 tons of the residue has been processed in solvent extraction equipment, resulting in the recovery of 377 g of neptunium. As part of the feasibility study for long-term neptunium processing, individual batches of the material were processed to determine the distribution of neptunium in the Paducah feed-plant fluorination equipment. The cyclone separator residues contained 66% of the neptunium in only 17% of the total ash.

In an effort to reduce the thorium content of the neptunium product solution, a flowsheet was developed and was demonstrated on one batch of neptunium product solution. The extractant for neptunium was 5% tributyl phosphate, and the primary salting agent was 8.0 M HNO_3. More than 99% of the neptunium was recovered, with separation from thorium by a factor of 240. (AEC Activity 2350)

Fluorox Process. — In further evaluation of the results of runs in the 3-in.-dia fluidized bed reactor, the total material balance was 95.9%, and UF_6 recovery in the cold traps and chemical traps was 72.9% of the theoretical amount. The two reaction rate constants determined for this run, 18.6 and 32.9 hr^{-1}, are somewhat higher than the extrapolated values from other sources. (AEC Activity 2702)

Dissolver Solution Analysis. — Evaluation of the performance of the ORNL transistorized controlled-potential coulometer has been completed; the instrument performs both oxidation and reduction titrations with excellent precision and accuracy.

Precision studies of the titration of uranium in sulfate medium were made in order to determine the loss of precision incurred by remote operation. The following relative standard deviation values were obtained for uranium at the

[redacted]

5-mg level: 0.1% when the titration was made in normal bench-top fashion; 0.2% when made in the ORNL High-Radiation-Level Analytical Facility with the ORNL remote pipettor; and 0.3% when the sample was run through an extraction procedure prior to titration, the entire operation being done remotely.

A search is now under way for the most advantageous separation procedure to be used in conjunction with the coulometric titration of uranium in various dissolver solutions. Ion exchange and solvent extraction techniques are being considered. The extractant triisooctylamine in xylene offers particular promise, since it is reported to quantitatively separate uranium from a large number of contaminants, and was found to cause little interference in the coulometric titration. The use of this extractant would obviate lengthy steps to remove organic material. Proper choice of electrolyte and potential in the subsequent titration may allow circumvention of any interference from substances not removed by the extractant. (AEC Activity 2724)

PROGRAM 3000 — WEAPONS

Special Separations. — A second high-purity Pu^{240} run was completed. The feed consisted of 1 g of 72% Pu^{240} recovered from the preceding high-purity run. An M-16 ion source and a single-pocket receiver were used in this separation, which yielded 39.1 mg of 99.5% Pu^{240}.

Two Pu^{241} separation runs provided 21.6 mg of 96.6% and 5.1 mg of 96.4% product. Both runs were made from a single 55-g charge of $PuCl_3$ containing 3.9% Pu^{241}.

Separation runs producing enriched U^{235} and U^{236} for second-stage feed continue. Very high purity U^{238} is being produced in three calutron tanks from Super-Q feed material.

Eighteen shipments of heavy-element isotopes were made: one U^{234}, two U^{235}, one U^{236}, one U^{238}, two Pu^{239}, four Pu^{240}, one Pu^{241}, and six Am^{241}. (AEC Activity 3610)

HOMOGENEOUS REACTOR PROJECT

In-Pile Slurry Loop. — Assembly of the loop is about half complete at the Westinghouse PAR Development Laboratory. The facility at ORNL, where the loop will be installed, was designed, and shielding and equipment cubicles for this facility are about 10% complete. (AEC Activity 4103)

Homogeneous Reactor Test. — The HRT was pressurized to 1700 psig and heated to 185°C on May 31, to study further the equilibrium mixing between core and blanket with dilute UO_2SO_4 in the system during operation as a single-region reactor. Tests were made in which the rate of condensate purge to the blanket was held constant at 6, 4, and 2 lb/min, and the equilibrium core and blanket concentrations at these conditions were determined by chemical analyses. The rate of intermixing was substantially constant at 3 to 4 lb/min. The ratio of the uranium concentration in the core to that in the blanket was approximately 3:1 at a blanket purge rate of 6 lb/min. The advantage of maintaining this concentration difference is that the power generation in the core can be kept high (60% of total). On the basis of these tests it was concluded that no modifications were necessary for reactor operation to continue. On June 4 the reactor was brought critical at an average temperature of 260°C, and the power was raised until the system output balanced the heat losses. Since that time the reactor behavior has been explored at core average temperatures of 240, 260, and 275°C and at total power outputs ranging from 250 to 3500 kw. The reactor has been circulating at temperature and pressure since May 30, accumulating over 800 Mwhr. (AEC Activity 4103.3)

HRT Chemical Pilot Plant. — The HRT Chemical Plant hydroclone loop was operated without incident for over 400 hr during two reactor operating periods. In 117 hr of operation with the reactor subcritical and at temperatures from 150 to 260°C, 158 g of corrosion products accumulated in the underflow receiver. System operation was normal during the first 300 hr of the third reactor power run. Reactor power was increased gradually from 400 kw to 2.5 Mw during this period. (AEC Activity 4103.1)

Fuel Processing. — Observations to date indicate that the removal of nickel by electrolysis in a simple cell offers little promise in the processing of HRT fuel. Conditions were sought under which nickel can be removed electrolytically from solutions simulating those expected in the outer dump tanks of the HRT (0.24 m UO_2SO_4, 0.12 m H_2SO_4, 0.18 m $CuSO_4$, 0.18 m $NiSO_4$). From such solutions cathodes of nickel or stainless steel gave better nickel deposition than a copper cathode gave; iron cathodes reacted and introduced iron into the solution. Since the deposition of nickel from such solutions onto the cathode must compete with other more favorable cathode reactions, such as reduction of uranyl ion to uranous, deposition of copper, and evolution of hydrogen, it is favored by high current densities at both cathode and anode, low solution temperatures, and use of a stationary cathode. At a cathode current density of 0.5 amp/cm^2 the rate of nickel deposition was not

greatly affected by varying the copper concentration between 0.18 and 0.007 m in 0.25 m UO_2SO_4, but was decreased by the absence of copper or by decrease of the nickel concentration. (AEC Activity 4103.1)

Uranyl Sulfate Blanket Processing. — Contaminated titanium metal that had been descaled with nitric acid containing a trace of hydrofluoric acid adsorbed plutonium from 1.4 m UO_2SO_4 at 250°C under oxygen pressure in a manner similar to that found in a new titanium container. Of 66 mg of plutonium (per liter) added in small increments, 75% remained in solution, 9.5% was adsorbed, and the remainder appeared as loose PuO_2.

Plutonium sorption on Dowex 50 (16% cross-linked) resin from a flowing stream of 1.4 m UO_2SO_4 containing 20 mg of plutonium per liter was at least 1.5 mg of plutonium per gram of resin with no appreciable plutonium breakthrough. (AEC Activity 4103.1)

Thorium Oxide Blanket Processing. — The amount of uranium adsorbed on 1600°C fired ThO_2 was decreased from 40,000 to <200 ppm by leaching at the boiling point with 4 N HNO_3 containing 1.5% H_2O_2 for 0.5 hr, apparently as the result of actual dissolution of the surface of the thorium oxide. A method for controlling the amount of ThO_2 dissolved has not yet been developed. When 1600°C fired ThO_2 was leached, ~2% dissolved. When 650°C fired ThO_2 was leached, 90% dissolved. (AEC Activity 4103.1)

Thorium Oxide Slurry Development. — A total of 525 lb of thorium oxide was calcined to 1600°C. Of this quantity, 240 lb was classified, recalcined at 650°C, and shipped; an additional 120 lb was classified. Thirty-five pounds of Houdry spheres was classified, recalcined, and shipped, and about 430 lb of unclassified 650°C calcined oxide was shipped.

In oxide preparation studies, flowsheets were developed for the pilot-plant production of 1- to 2-μ average size cubical thorium oxide particles and ~2.5-μ average size thorium-uranium oxide particles containing 0.5% uranium. A flame calcination apparatus was satisfactorily operated with methyl alcohol slurries of $Th(C_2O_4)_2$ and ThO_2. In duplicate runs, results were reproducible, with ThO_2 surface areas of 2.0 and 1.2 m^2/g being obtained with carbon and ZrO_2 reflectors, respectively.

Circulating ThO_2 slurries were simply and efficiently collected in an induced underflow receiver by 0.4- and 0.8-in.-dia hydroclones. Results of studies of hydroclone performance for concentrated ThO_2 slurries (~100 g of ThO_2 per liter) showed that classification of 0.5- to 5-μ-dia ThO_2 is practical and that preparation of clarified purge water by a simple cascade with one pump may be practical.

In a 3000-hr irradiation of a settled slurry of 1600°C fired ThO_2 in the LITR, corresponding to a buildup of mass-233 isotopes to ~0.2%, no gas pressure in excess of steam pressure was observed. A slurry (250 g of Th per kg

of D_2O) of 1000°C fired thorium-uranium oxide (U/Th = 0.05) containing no gas recombination catalyst showed a radiolytic gas pressure of 60 psi at 280°C when irradiated in the LITR. In out-of-pile studies with a similar slurry containing 0.02 m MoO_3, the D_2 reaction rate was about 6.0 moles/hr per liter of slurry at 280°C and a partial pressure of 100 psi of D_2 for a slurry that had been activated by heating with D_2. Similar slurries to which no MoO_3 was added developed substantial catalytic activity (>2 moles of D_2 per hr per liter at a pressure of 100 psi of D_2) when heated with D_2. (AEC Activity 4103.1)

HRP Analytical Chemistry. — Microgram and submicrogram quantities of copper, lead, and cadmium in uranyl sulfate solutions were determined by a modification of the anodic-stripping polarographic method of Nikelly and Cook (Anal. Chem. 29, 933, 1957). Similarly, cathodic rather than anodic stripping was utilized in the estimation of chloride in the range of 0.1 to 1 μg/ml with a coefficient of variation of the order of 15%. In this application the stripping technique of Zittel *et al*. (ORNL-2453) was used; however, a high-sensitivity polarograph was substituted for the electronic coulometric stripper utilized by Zittel.

A radio-frequency method of the type described by Stelzner and Kelley (ORNL-1742) is being used in the titration of 50 to 75 mg of thorium with oxalic acid or ethylenediaminetetraacetic acid (EDTA). The end point is established by plotting the oscillator grid current or the IR drop across a load resistor vs the volume of titrant and observing the break in the curve. The coefficient of variation is about 0.7%. Interference by substances which obscure the end point in visual titrimetry can be avoided by the use of this method.

The fusion-pyrolysis method, described previously for the separation of fluoride from thorium oxide and other refractory materials (ORNL-2512), is being utilized for the separation of microgram quantities of nonleachable chloride from high-fired (1600°C) thorium oxide. Sodium molybdate is used as a flux instead of sodium bismuthate, which was used for the removal of fluoride. (AEC Activity 4103.1)

Slurry Testing and Evaluation. — The object of the testing and evaluation program is to develop a slurry which has long-term resistance to degradation under reactor operating conditions, is easily resuspended from settled beds, exhibits low attack on structural materials, and approaches Newtonian fluids in flow characteristics (i.e., has a low yield stress). The best thoria presently available is composed of particles with diameters in the range 1 to 2 μ and fired to 1600°C. However, the effect of reactor additives (uranium, molybdenum oxide for gas recombination, and fission products) on the rheological properties must be evaluated. Such evaluation is being carried out in a series of loop circulation runs at 280 to 300°C, of which run 200-A-15 is typical. The run has continued for over 2800 hr, during which time uranium and molybdenum oxide have been added to the thoria. The attack rate on stainless steel has dropped to about 0.6 mil/year, and the slurry has remained stable.

In an effort to furnish data for optimization of yield stress and attack rate with respect to particle size and shape and appropriate chemical treatment, seven runs in 100-gpm loops were completed in which the effects of long-term oxalate digestion (prior to conversion to ThO_2) on 800°C fired oxide, of 300 ppm SO_4 on 1600°C fired oxide, and of increasing particle size (average 3 μ instead of 1 to 2 μ) were investigated. An eighth run is in progress wherein the thoria particles have been coated with SiO_2 in order to produce an exceedingly fluid slurry. A large number of additional runs are required before conclusions can be drawn or a superior composition selected. (AEC Activity 4103.1)

Slurry Blanket System Development. — The 300-SM blanket test facility is operating with a slurry whose concentration is 323 g of Th per kg of H_2O for final measurements of circulation in a blanket of HRT geometry. This information was developed to a large extent in the previous run (SM-4), in which high-velocity inlet nozzles were used; the present run will indicate the influence of nozzle velocity on circulation and thoria concentration patterns, inasmuch as lower-velocity HRT-type nozzles are currently installed.

The run was started on May 2, 1958; from May 21 to May 27, 525 kg of thoria was charged. From the results of systematic sampling it is concluded that there has been no significant concentration gradient in the blanket, although a decrease in concentration is indicated by gamma transmission measurements both at the very top and at the bottom of the blanket. The circulating concentration in the loop is somewhat lower than the average blanket concentration, as was true in previous runs.

The new blanket vessel being fabricated by Combustion Engineering has been forged and is undergoing machining. A spare head closure has been designed to permit use of the vessel either as a small-scale blanket model of a two-region reactor or as an intermediate-scale model of a single-region homogeneous slurry reactor of about 5 Mw power. (AEC Activity 4103.1)

Blanket Engineering Research. — The patterns of fluid flow in a cylindrical vessel having a concentric inlet and outlet, representing a possible reactor core design, were studied by observing the motion of phosphorescent particles activated by a plane light source. The inlet pipe had to extend to within three core diameters of the vessel bottom before a well-defined fluid jet was obtained which swept the bottom.

The accuracy of the Bingham relation for describing the plastic flow characteristics of thoria slurries was investigated by applying it to data obtained with the horizontal capillary-tube viscometer. The Bingham relation was found to be adequate for shear rates in the ranges of interest.

The first slurry run in the 30-gpm loop resulted in the formation of thoria spheres and cakes. This behavior duplicated results obtained previously with the same slurry in a 100-gpm loop.

Tests associated with fluidizing Houdry thoria spheres in a 4-in.-dia column showed that the particles attached themselves to air bubbles in the water and were slowly carried out of the column (1000 g in 5 days). This behavior was not observed when "standard slurry" spheres were fluidized. (AEC Activity 4103.1)

Core and Blanket Vessel Development. — Tests conducted on a full-scale HRT core flow model with sand and water demonstrated that suspended solids did not leave the core but collected on the screen diffuser and the walls. A full-scale model of a replacement HRT core has been installed in a test system in order to study the behavior of suspended solids, the heat transfer conditions at the core wall, and the general flow pattern.

The 30-in. low-pressure blanket model is being converted to a slurry-core model. Fabrication is 85% complete.

Tests of a jet pump proposed to amplify the flow through a blanket vessel showed that increases in apparent throughput by a factor of 2.6 could be achieved. (AEC Activity 4103.1)

Centrifugal Circulating Equipment. — Considerable difficulty was experienced in shakedown runs with water in the 300A slurry loop as a result of the carrying of bubbles from the gas pressurizer into the circulating stream. This condition was corrected by reducing the flow of water through the jet liquid-vapor contactor, which is part of the pressurizer design, to 12 gpm. The Koppers floating bushing shaft seal of the 300A pump appeared to prevent upward mixing of fluids from the pump into the motor in these preliminary runs.

Conversion of the 400A-2 loop and pump into the 200Z slurry system was initiated. (AEC Activity 4103.1)

Miscellaneous Equipment. — Satisfactory welding tests were conducted on the 3 1/2-in. titanium 110AT pipe which will be applied to a boiling uranyl sulfate pressurizer.

Methods for locating and viewing the break in the HRT core tank were investigated on a full-scale mockup of the HRT core and pressure vessel. It was demonstrated that the periscope arrangement used at the reactor did not permit the viewing of the entire conical portion of the core tank. This periscope was modified to increase its range and improve the lighting; a second periscope was fabricated which should correct the difficulties experienced with the first model. A television camera manipulator was fabricated and will be tested. Methods of verifying the presence of cracks with dry ice fog and dye were demonstrated. (AEC Activity 4103.1)

Heat Removal Equipment. — The replacement HRT heat exchanger was received from Babcock and Wilcox and was subjected to 25 thermal cycles between 380 and 580°F. The unit showed no detectable leakage in hydrostatic and helium leak tests following thermal cycling. (AEC Activity 4103.1)

Gas Handling Equipment. — The solubility of the stoichiometric mixture of hydrogen and oxygen in a solution of uranyl sulfate in light water was found to be about 1.8 times that predicted from the individual gas solubilities at 250°C. This measurement is tentative and is being checked carefully.

Syracuse University reported that the no-reaction limit for the stoichiometric mixture of hydrogen and oxygen in the presence of steam is 6 mole % of gas. The corresponding limit in a series of runs with deuterium and heavy water was 30 mole % of gas, which indicates that deuterated vapors are considerably safer. The lower detonation limit was also higher in the deuterated system. A test in which uranyl sulfate solution was boiled in titanium pipe at 1500 psi was completed successfully after 1600 hr at the 0.04 m level and 1000 hr at the 0.17 m level. (AEC Activity 4103.1)

Fuel Systems Development. — The HRT fuel mockup system is being used to test improvements which can be incorporated in the HRT and to explore conditions encountered in HRT operation which are connected with unanticipated performance or difficulties.

Run 22 was completed May 22, 1958. Its primary purpose was to measure the corrosion resistance of a titanium letdown heat exchanger. The generalized system corrosion rate when circulating HRT fuel (using natural uranium) was only 0.8 mil/year, or half the value previously experienced with a stainless steel heat exchanger, thus indicating the superiority of titanium in the sensitive temperature range for stainless steel.

The precipitation of fuel in the pressurizer-to-system connecting line, encountered in the initial HRT criticality runs, was found to be reversibly dependent on the combination of excess fuel acidity and the condensate purge flow to the pressurizer (used to prevent uranium from entering the boiling region). For example, the purge flow must be reduced to 2 gph to redissolve uranium if the excess acidity is no greater than 55%, and a 4-gph purge flow requires 72% excess acidity to prevent precipitation.

Excessive transport of fuel to the rear of the HRT circulating pump can reduce the motor life by increasing the radiation exposure of the windings. The transport, due to mixing of fuel and condensate purge across the shaft seals, has been measured in connection with studies of motor winding life. During March and April the mixing rate was between 2.7 and 3.6 liters/hr. However, the rate decreased steadily during the last 400 hr of the run to a value of only 0.04 liter/hr. No explanation has been found for the change. (AEC Activity 4103.1)

Feed Pump Development. — The life prognosis for HRT fuel feed pumps and purge pumps has been increased to greater than 10,000 hr as a result of continued satisfactory operation of five test heads.

A direct water drive for a diaphragm pump, which would eliminate the requirement for an intermediate system such as is used in the HRT, was operated. The system performed well, but the life of the water cylinder rings was unsatisfactory.

The one-stage turbine pump was fitted with an electrolyzed type 316 stainless steel impeller and aluminum oxide wear rings. It has since delivered 10 gpm of fuel solution at 300 psi for 1100 hr with no change in characteristics. If the test continues to be successful, it would indicate the feasibility of a multistage turbine feed pump.

Because of the short life experienced to date with HRT letdown and dump valve trim, two new valves were designed and were issued to the shop for construction. The letdown valve throttling is accomplished by means of five orifices formed by a series of 5/8-in. balls in tapered barrels. The dump valve trim consists of an aluminum oxide ball and seat.

Tungsten carbide—trimmed hand valves are being tested for various slurry shutoff and throttling applications. Their performance to date has been greatly superior to that of the metal-trimmed valves used formerly. (AEC Activity 4103.1)

Gaseous-Fission-Product Disposal. — A survey to determine the relative efficiencies of various solid adsorbents for removing rare gases from a flowing stream of oxygen was completed. The materials were compared on the basis of adsorption of Kr^{85} from a flowing oxygen stream at 25°C. The most effective adsorbent is charcoal, with Columbia grade G activated charcoal superior to other charcoals tested. Linde molecular sieve materials (5A, 10X, 13X) are 20 to 40% as effective as charcoal. Silica gels are 10 to 15% as effective as charcoal.

The effect of diluent gas composition on the adsorption of Kr^{85} by Columbia G charcoal was studied. The adsorption was most efficient when helium was the diluent gas, and was lower by a factor of 3 when helium was replaced by oxygen, nitrogen, argon, or air.

Equilibrium adsorption isotherms for Kr^{85} on Columbia G charcoal gave a value of 0.0045 mg of Kr per gram of charcoal at 80°C and 0.1 mm krypton partial pressure, compared with 0.0296 mg of Kr per gram of charcoal at 28°C. Standard carbon wool (type B-6) at 28°C and 0.1 mm krypton partial pressure had a capacity of 0.0162 mg of Kr per gram of wool, or 55% of the capacity of Columbia G charcoal. Alcoa activated alumina H-151 had 3.7% of the Columbia G charcoal capacity under the same conditions. (AEC Activity 4103.1A)

MOLTEN-SALT REACTOR (MSR) PROGRAM

Molten-Salt Reactor: Design. — A study was made of a circulating-molten-salt-fueled reactor power plant which would operate at 5 Mw without a fuel pump (circulation by thermal convection) or at 50 Mw with a fuel pump. This system provides for the development of nuclear and engineering data in a full-scale reactor system at low power density and for the extension of the data to higher power density in the same reactor.

A Loëffler boiler cycle, which has only the superheater in contact with the heat source, was investigated for removing heat from a molten-salt reactor. It was found that, either with or without an intermediate circuit (such as sodium) for thermal linkage, the Loëffler boiler cycle would provide a differentially controllable thermal block between the fuel and water. Only conventional steam- and water-handling components would be required for effecting complete control of the reactor and the load relationship.

Machine calculations on two-region molten-salt reactors fueled with U^{233} and Pu were continued. The U^{233}-fueled reactors show considerable promise, especially in the larger sizes. Comparative data for U^{233}-fueled systems with 339 ft^3 of fuel external to the core and 4 mole % ThF_4 in the fuel are given below:

Core diameter, ft	6	8	10	12
Critical mass, kg of U^{233}	149	246	406	479
Critical inventory, kg of U^{233}	595	559	670	869
Regeneration ratio	1.013	1.025	1.043	1.038

Similar data for Pu-fueled systems with no ThF_4 in the fuel show the following:

Core diameter, ft	6	8	10
Critical mass, kg of Pu	19.7	13.7	16.3
Critical inventory, kg of Pu	78.1	31.1	24.2
Regeneration ratio	0.463	0.352	0.272

(AEC Activity 4103.1B)

Molten-Salt Reactor: Experimental Engineering. — In the development of remote maintenance facilities for molten-salt reactors, it was found that leak-tight indented-seal joints could be formed by using a soft-iron nickel-plated gasket and tightly clamping the flanges. A frozen seal formed in the annulus between the flanges during testing in a molten-salt system. The feasibility of making critical welds remotely by using a pair of manipulator arms and Heliarc welding equipment was demonstrated. A prototype combination heater and insulation unit suitable for remote installation and removal was fabricated for testing. Components and facilities are being designed and procured for a remote maintenance demonstration facility.

A small molten-salt pump that operates submerged in the pumped fluid was built for testing. The shaft is sealed with frozen lead supplied from a reservoir within the pump. The pump is operating isothermally at 1250°F, and there has been no leakage. (AEC Activity 4103.1B)

Molten-Salt Reactor: Radiation Damage. — Two low-permeability graphite capsules filled with $LiF-BeF_2-UF_4$ (62-37-1 mole %, fuel 130) and enclosed in Inconel containers were inserted in the MTR for irradiation at 1250°F at a power density of 1200 w/cm^3. These capsules, which were inserted in the MTR on May 28, 1958, are to be irradiated for an indefinite, but lengthy, period. (AEC Activity 4103.1B)

Molten-Salt Reactor: Metallurgy. — Embrittlement studies of INOR-8 aged for 2000 hr in the temperature range 1000 to 1400°F were completed. No significant change was noted in the room-temperature tensile properties of the aged material.

Relaxation tests were completed on INOR-8 at 1400 and 1500°F and are in progress at 1150 and 1175°F. It has been observed that some time elapses before creep begins.

Creep tests of INOR-8 in salt environments were run at 1100, 1200, and 1300°F and at stresses ranging from 12,000 to 30,000 psi, and creep rates for these conditions have been established. Consistency tests show excellent correlation for INOR-8 creep rates.

The tensile properties of INOR-8 weld metal were determined at room temperature, 1200, 1300, and 1500°F. A lowering of ductility was observed at 1500°F. Back-brazing techniques for use in the fabrication of INOR-8 heat exchangers with thick tube sheets were developed, and a 5-in.-thick header was brazed.

Commercially produced seamless 1/2-in.-OD x 0.045-in.-wall INOR-8 tubing was examined for adherence to dimensional tolerances and for defects. It was found to be well within commercial tolerances, and the amount of defective material located was comparable to that found in a good grade of Inconel.

Carburization tests of INOR-8 and Inconel specimens in sodium-graphite systems at 1200, 1400, and 1600°F for 40 and 400 hr were completed. No carburization occurred at 1200°F, but the carburization increased with the temperature and the specimens showed decreasing ductility.

Hot-leg sections of two Inconel forced-circulation loops operated for approximately 3000 hr with $LiF-BeF_2-UF_4$ fuels were examined. The maximum depths of void formation observed, 7 and 5.5 mils, were higher than those observed in thermal-convection loops. An Inconel loop operated with NaF-LiF-KF salts for 2700 hr had a maximum of 2 mils of void formation. An INOR-8 loop operated for 1300 hr with the NaF-LiF-KF mixture had no attack. (AEC Activity 4103.1B)

Molten-Salt Reactor: Chemistry. — Phase equilibrium studies of the system $LiF-ThF_4-UF_4$, which have been completed, showed that invariant equilibria occur for the following compositions and temperatures: 71.5 mole % LiF—7.0 mole % ThF_4—21.5 mole % UF_4, 500°C (peritectic); 72.0 mole % LiF—1.5 mole % ThF_4—26.5 mole % UF_4, 488°C (eutectic); 60.0 mole % LiF—22.0 mole % ThF_4—18.0 mole % UF_4, 605°C (peritectic). Because of the complete miscibility of the pairs ThF_4-UF_4, $LiF \cdot 4ThF_4-LiF \cdot 4UF_4$, and $7LiF \cdot 6ThF_4-7LiF \cdot 6UF_4$, no ternary invariant equilibria involve members of all three of these pairs of solid solutions.

The solubility of PuF_3 in $LiF-BeF_2$ and $NaF-BeF_2$ mixtures at temperatures in the range 565 to 650°C appears to be sufficiently high to provide a fuel system for a molten-salt plutonium-burner reactor. In an $LiF-BeF_2$ mixture with 63 mole % LiF, the solubility reaches a minimum of 0.23 mole % PuF_3 at 565°C, while in the $NaF-BeF_2$ system (57 mole % NaF), a minimum of 0.18 mole % PuF_3 is reached. The addition of 1.0 mole % ThF_4 to the mixture $LiF-BeF_2$ (62-37 mole %) had no significant effect on the solubility of PuF_3. The addition of CeF_3 to the same solvent mixture significantly reduced the solubility of PuF_3. Removal of PuF_3 for reprocessing purposes by dissolving CeF_3 in the mixture at a high temperature and filtering out the precipitated CeF_3-PuF_3 solid solution at a lower temperature should prove to be possible.

A rather satisfactory correlation of the solubilities of noble gases in molten fluorides was obtained from the relation

$$K_c = e^{-18.1r^2\gamma/RT},$$

where K_c is the ratio of the concentrations of gas in solution and in the gas phase, r is the atomic radius of the noble gas in angstroms, γ is the surface tension of the solvent in dynes/cm, R is the gas constant in $cm^3 \cdot atm \cdot mole^{-1} \cdot (^oK)^{-1}$, and T is the temperature in °K. This relation can be readily obtained if it is assumed that the entropy of solution is negligible and that the internal energy of solution is due to the formation in the molten salt of spherical holes of the same size as the gas atoms. The solubilities calculated by means of the above equation agree with the measured solubilities in all cases to within a factor of 10 and often much better.

Analytical results from corrosion tests with chromium metal in 29 mole % KCl—71 mole % $ZnCl_2$ show that the corrosion of Inconel, where the activity of chromium is about 0.1, would become very serious after long contact. The mixture 58.3 mole % LiCl—41.7 mole % RbCl, which appears to offer more hope from the corrosion standpoint, was found, after contact with chromium metal for 72 hr in fused silica, to contain 300 ppm of chromium, which represents the solubility of Cr_2O_3 resulting from the reaction.

$$6LiOH + 2Cr^o \longrightarrow Cr_2O_3 + 3Li_2O + 3H_2.$$

(AEC Activity 4103.1B)

Molten-Salt Reactor: Heat Transfer and Physical Properties. — Studies of the hydrodynamics of spherical cores proposed for a molten-salt power reactor were continued with an investigation of the flow in a core with a concentric entrance-exit system. The central, or exit, pipe was flared outward so that the fluid entering through the annulus was directed along the surface of the sphere. The flow patterns, indicated by observation of particle motion in an illuminated plane, showed the nature of the fluid motion in this model to be dictated by the manner in which the fluid entered the annulus, rather than by the annular entrance per se. Thus the fluid, which entered the annulus through a single side arm about one pipe diameter above the core entrance, flowed past the central pipe and generated, within the core, full-volume circulation with flow down along the sphere surface directly opposed to the side arm and up along the opposite surface. It was further observed that this circulation extended into the exit pipe and that some of the flow recirculated through the core. Several static regions were also noted.

Preliminary values were obtained for the viscosities of two additional beryllium-containing fluoride mixtures. For the composition $NaF-BeF_2-UF_4$ (53-46-1 mole %), the viscosity varied from 15 centipoises at 600°C to 4 centipoises at 800°C; somewhat higher values, 21 centipoises at 600°C to 6 centipoises at 800°C, were obtained for the mixture $LiF-BeF_2-UF_4$ (53-46-1 mole %). The heat capacity of the mixture $LiF-BeF_2-UF_4$ (62-37-1 mole %) was found to be $(0.488 + 0.00005\ T)\ cal \cdot g^{-1} \cdot (^{\circ}C)^{-1}$ over the temperature range 471 to 800°C. (AEC Activity 4103.1B)

Molten-Salt Reactor: Fuel Processing. — Three runs were completed in studying the feasibility of removing uranium from $LiF-BeF_2-UF_4$ (47.6-51.6-0.8 mole %) fuel by fluorination. In 2.5-hr fluorinations the uranium losses to the waste salt at 550, 500, and 450°C were, respectively, 1, 2, and 5% of the initial amount. The induction period observed with $NaF-ZrF_4$ was not observed with $LiF-BeF_2$.

In further tests on the dissolution of $LiF-BeF_2$ (63-37 mole %) in aqueous HF solutions, it was found that crushed (but not powdered) salt dissolved rapidly in 90 wt % HF, with equilibrium concentrations of 70 g/kg for both LiF and BeF_2; the concentration reached 50 g/kg for both in 15 min. At higher HF concentrations BeF_2 was less soluble; at lower concentrations LiF was less soluble. In solutions saturated with LiF, the BeF_2 solubility appeared to level off at 25 to 30 g/kg as the HF concentration approached 100%; in the absence of LiF it decreased to near zero.

The solubility of rare-earth fluorides in $HF-H_2O$ solutions decreased from 0.026 to 0.008 g/kg as the HF concentration increased from 80 to 98 wt % (0.0034 to 10^{-4} mole % based on dissolved $LiF + BeF_2$). The reactor design calls for a steady-state concentration in the core of about 0.05 mole % rare earths.

In general, 70% of the strontium tracer activity associated with the dissolved $LiF-BeF_2$ salt was found in solution. All the cesium in the $LiF-BeF_2$ salt dissolved upon equilibration with the HF solutions, including the cesium in the undissolved excess salt. (AEC Activity 4103.1B)

CIVILIAN-POWER ADVANCED GAS-COOLED REACTOR (GCR)

Gas-Cooled Reactor. — A decision was made that ORNL is to be responsible for the procurement of the fuel elements for the gas-cooled reactor which is to be built by Kaiser-ACF for the AEC. It was further established that the reactor is to be helium-cooled. The research and development work necessary to establish procedures for the fabrication and inspection of the stainless-steel-clad enriched UO_2 fuel elements is under way. Facilities for tests of the fabrication and assembly procedures are being designed. A survey is being made of facilities for in-pile testing of materials in order to determine the effects of radiation on mechanical properties. Such tests will include, ultimately, tests of a full-size fuel element under simulated operating conditions. Fabricators are to be contacted and steps are to be taken to establish the liaison and inspection routines that will be required during the final production of the initial core loading. ORNL personnel will also review the Kaiser-ACF design as it proceeds and will provide technical assistance as required.

Two more 1000-hr tests of TSF graphite and various steels in a helium environment at 1400°F were concluded. In one of these tests the helium was static, and in the other the helium circulated by thermal convection. No effect of the circulation of the helium could be detected. Examinations of the thin surface films detected on the specimens have not yet been completed. A further test was initiated.

In the nondestructive testing studies it was established that defects 0.001 in. deep and 1/8 in. long could be detected on the outer surface of 3/4-in.-OD, 0.020-in.-wall stainless steel tubing with the eddy-current instrument and coil system.

Irradiation of the first in-pile capsule was completed on June 17. Specimens of 15% enriched UO_2, which generated a power density of ~750 w/cm^3, were irradiated in the LITR at a UO_2 surface temperature of 1500°F and a central temperature of 1770°F. As stated previously, these specimens were irradiated primarily to obtain data on the release of fission-product gases, but approximately the same thermal stresses as those anticipated in a fuel element were present, and therefore information on the integrity of UO_2 under reactor conditions will be obtained. Upon removal from the reactor, the sealed ampoule was punctured and several samples of gas were extracted. The gas is now being analyzed for the amount and kind of fission products. Equipment was prepared for irradiation of full-size stressed UO_2 pellets. Tests will be initiated as soon as the pellets become available. (AEC Activity 4119)

MARITIME SHIP REACTOR PROGRAM

Maritime Ship Reactor. — The Laboratory continued to furnish technical assistance to the Maritime Reactors Branch (AEC) in the program to develop nuclear-powered merchant ships. Review of drawings and specifications for the NS "Savannah" reactor system (NMSR) continued, with particular attention being given to reactor safeguards analyses, containment specifications, and primary shield design. Calculations were made of the activities to be expected in the cooling water and in the demineralizer.

Brazed joints similar to those in the NMSR fuel bundle were made and tested. The results indicate that the proposed brazing alloy, Nicrobraz 50, should be satisfactory as a joining agent for the fuel bundles. In order to evaluate inspection techniques for welded joints in fuel-tube end closures, several specimens were prepared according to each of three end closure designs. All joints were found to be leak-tight in a test with helium and a mass spectrometer. X-ray examinations indicated that each type of joint could be economically examined for defects. Radiographic inspection procedures were developed.

A preliminary bill of material was issued for the pressurized water loop which will be installed in the Oak Ridge Research Reactor. Specifications for most of the major components were written, and procurement was initiated for the pumps and for parts of the makeup system and instrumentation. A design layout of the in-pile section was made. Instrumentation schematics and structural design are nearly complete.

Studies continued toward the development of criteria for the health physics monitoring program for the NS "Savannah." An outline for the procedure manual was prepared.

In the waste disposal study, a mixed resin bed containing sorbed fission products was homogeneously mixed in portland cement, and the leaching effect of sea water was measured. After 17 days an equilibrium concentration was apparently reached in which 8.2% of the total gross gamma and 2.6% of the gross beta activities appeared in the sea water. (AEC Activity 4151)

PACKAGE REACTOR DEVELOPMENT

Army Package Power Reactor (APPR-1). — Metallurgical experiments were conducted to evaluate the use of prealloyed type 304L stainless steel containing enriched B^{10} as a vehicle for incorporating burnable poison in the fuel plates for core II. Sample fuel compacts whose ratio of surface area to volume was 1.2 were found to evolve approximately one-third the boron content during sintering. Increasing the ratio of area to volume increased the boron loss during this operation. Because of the present uncertainty as to the retention of boron within the alloy, a tentative decision was made to use B_4C as the burnable poison additive in a procedure similar to that used for core I.

Boron carbide containing partially enriched B^{10} is being procured as a possible means for increasing the B^{10} content of core II without increasing the amount of B_4C.

Stainless steel of the 304L type but with the cobalt content reduced to 0.005% was received for use in the fabrication of core II. Preliminary fabrication experiments indicated no significant difference in the fabrication properties of this material as compared with those of ordinary type 304L stainless steel. (AEC Activity 4601)

Army Gas-Cooled Reactor Development. — The hazard report for the Army gas-cooled reactor experiment (GCRE) was reviewed at the request of the Army Reactor Branch (AEC). Comments on the report were submitted to the AEC.

The entire primary system of the GCRE was simulated on the ORNL analog computer. The system was perturbed by simulating loss of flow, heater failure, changes in moderator temperature, and small step changes in reactivity. The temperatures of the fuel plates and gas coolant and the power levels were recorded as a function of time. Preliminary results indicate that the control system should be designed to act slowly enough not to obscure fast-period information resulting from an accident, since the fast-period information is needed for the fast safety system. (AEC Activity 4603)

GENERAL REACTOR RESEARCH

Basic Reactor Research. — A calculation of the maximum temperatures to be expected in the stainless-steel-clad BSR-II fuel elements if the reactor should be shut down by loss of the cooling water was carried out by extrapolating the results of an experiment performed with the BSR-I. The BSR-I experiment was used to find heat transfer parameters by two different models for convective heat transfer. These parameters were then used to calculate the BSR-II temperature due to coolant loss as a function of time after shutdown by means of step-wise approximations to the heat generation and heat loss equations. The maximum temperature was found to be well below the danger point for melting of stainless steel.

The new BSF accelerator in Building 3010 was completed and was successfully tested without pulsing. The rated high voltage of 300 kv was attained, and well-focused beams up to 150 μa were easily observed. This accelerator will be used in the experiment to determine the neutron diffusion parameters of various media. (AEC Activity 4202)

Fuel Element Development. — Development of aluminum plate-type fuel elements containing 20% enriched uranium in the form of a 40 to 50 wt % U—Al alloy modified with a 3% Si addition has been essentially completed. The utility of uranium carbides for this type of application has also been determined. Specimens containing both the silicon-modified alloy and dispersions of uranium dicarbides in aluminum have been fabricated for irradiation testing in the MTR.

Preparations have been completed for manufacturing a 20% enriched Mark XI MTR fuel element containing a 60.5 wt % dispersion of U_3O_8 in aluminum for irradiation testing in the active lattice of the MTR. A dry run resulted in successful manufacture of a fuel element containing normal U_3O_8. Oxide containing 20% enriched uranium has been received, and composite plates containing this material have been fabricated.

Additional data on the diffusion of uranium through the aluminum cladding of composites containing U-Al alloys after 6 hr at a temperature of 600°C support the previous 3-hr data, which revealed an increase in the level of uranium at the plate surface after the heat treatment. (AEC Activity 4203)

Power Reactor Fuel Reprocessing: Alcohol-Chloride Process. — In this process, currently being studied on a laboratory scale, U-Zr-Nb alloy fuels are dissolved in alcoholic HCl or $HgCl_2$ solutions. The residual mercury is reduced to metal electrolytically and separated, HNO_3 is added, and the HCl is distilled off to produce solvent extraction feed. Recovery of mercury for recycle from spent $HgCl_2$-ethanol dissolving solutions by an electrolytic process was demonstrated, with losses of less than 0.1%. The mercury was converted to the soluble mercuric ion by anodic oxidation and then deposited as mercury at a mercury-pool cathode in a cell operated at 50 to 80°C at a current density of 1 to 1.5 amp/cm^2. (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Alternate Aqueous Dissolution. — A tentative flowsheet has been developed for the decladding of PWR blanket rods with boiling 6 M NH_4F—1.0 M NH_4NO_3. The UO_2 core is then dissolved in 10 M HNO_3 to form a solvent extraction feed solution. The total uranium loss with unirradiated nonoxidized fuel specimens was less than 0.022%.

Several modifications of the NH_4F-HNO_3-HF flowsheet for EBWR core 1 fuel are being investigated. Complete dissolution in NH_4F, followed by metathesis with NH_4OH and subsequent dissolution of the solids in nitric acid, appears preferable, since niobium is completely soluble in the nitric acid solution without the addition of large amounts of fluoride. Decladding of the fuel in NH_4F, followed by direct addition of HNO_3-$Al(NO_3)_3$ solution to dissolve the core, resulted in precipitation of niobic acid soon after core dissolution started. Addition of hydrogen peroxide provided temporary dissolution of the precipitate when the uranium concentration was less than 0.3 M. At higher uranium concentrations, addition of H_2O_2 did not prevent precipitation.

The use of mixed nitric and hydrofluoric acids appears unattractive for the total dissolution of Consolidated Edison fuel. In 1 M HF—1 M HNO_3, type 304L stainless steel dissolved at only 0.82 mil/hr in a 6-hr test. (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Materials Evaluation. — In Sulfex-Thorex tests, Carpenter 20Cb was corroded in 20 cycles at an over-all rate of 1.57 mils/month when the cycle consisted of alternate treatments with 6 M H_2SO_4 and the final Thorex dissolver solution. When the cycle consisted of alternate treatments with 4 M H_2SO_4 and the initial Thorex dissolver solution, the over-all rate after 20 cycles was 2.34 mils/month. In 24-hr scouting tests, Nionel was corroded at rates of about 50 and 0.8 mil/month in the initial and final Thorex dissolver solutions, respectively, in the absence of Al^{+++}. The intergranular stress cracking of Carpenter 20Cb on exposure to sulfuric acid was at least partially decreased by heat treatment, and was inhibited by the presence of stainless steel dissolution products. No cracking was observed with Nionel under any test conditions.

Dimetcoat No. 3, a protective coating manufactured by Amercoat, and Amercoat No. 1649, a cover coat for Dimetcoat No. 3, were irradiated to 10^{10} r of gamma rays in water. Dimetcoat No. 3 showed no visible physical damage, while Amercoat No. 1649 became chalky and showed low abrasion resistance. Pfaudler's No. 53 glass on Inconel, on type 304L stainless steel, and on mild steel showed no visible physical damage when irradiated to 10^{10} r of gamma rays in either air or water. The irradiated specimens were returned to Pfaudler for physical property testing. (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Solvent Extraction Studies. — Flooding studies for the 42.5% tributyl phosphate—Decalin and water system were completed. A plot of log flooding rate vs pulse frequency was linear over the frequency range 50 to 80 cycles/min. (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 two hydrochlorination runs at approximately 900°C, 64.3% of one element reacted in 5.5 hr and 76.3% of another reacted in 5 hr. In both runs the HCl was passed through activated charcoal and was about 55% utilized. Uranium losses to the sublimate were 0.057%, and 5.1% was insoluble in 10 M HNO_3. (AEC Activity 4301)

Hermex Process. — In the Hermex process, uranium is dissolved in mercury or amalgam and recovered in the zero valence state. The solubility of uranium in 0.1 wt % Mg(Hg) was greater by a factor of 1.5 than in mercury alone. The solubility increased from 0.0056 wt % at room temperature to 1.41 wt % at 356°C. A heat of solution of 5.9 kcal/mole was calculated. (AEC Activity 4303)

Fused Salt—Fluoride Volatility Process. — The feasibility of removing uranium from $LiF-NaF-ZrF_4$ was demonstrated in several fluorination runs. In fluorinations of 27-18-55 mole % salt at 600°C containing 4000 to 5000 ppm uranium, retentions were about 2 ppm, that is, about 0.05%. Test runs with 26-37-37 mole % salt were also successful, even at lower temperatures. Uranium retentions were less than 0.05% at 535 and 625°C under conditions otherwise the same. At 450°C, however, the uranium retention was 22 ppm, indicating

that the rate of the fluorination reaction begins to significantly decrease in this temperature range.

INOR-1 was corroded at a rate of 2.1 mils/month in 500 hr exposure to fused 50 mole % $NaF—ZrF_4$ in the presence of flowing HF at 650°C. INOR-8 showed 1.2 mils/month corrosion under the same conditions. (AEC Activity 4302)

Waste Treatment and Disposal. — In studies on more economical methods of treating radioactive wastes from irradiated fuel element processing, five standard waste types neutralized with NaOH or $Ca(OH)_2$ and evaporated to dryness had residual volumes 5 to 36% of the original waste volume, depending on the waste type and the base used.

Neutralizations of Darex process wastes with urea gave a granular precipitate that was easy to filter. Nickel was precipitated better by $Ca(OH)_2$, but the precipitate was gelatinous, slow settling, and slow filtering. When Darex waste was evaporated to 1/4 volume, the condensate was decontaminated by a factor of 10^4. The concentrate was viscous and solidified upon cooling. Under the same conditions except for the addition of formaldehyde as a denitrating agent, results were the same except that the viscosity of the concentrate was lower. Nitrate was almost completely destroyed when Darex wastes acidified with sulfuric acid were treated with formaldehyde.

The solids resulting from reducing Sulfex waste to dryness totaled 42% of the original weight. Sulfex waste neutralized with 50% NaOH formed a thick brown gel.

Doubling the amount of sea water used to leach mixed resins embedded in cement increased the amount of activity leached by only ~20%. (AEC Activity 4305)

Long-Range Planning Studies. — Studies have been made of the neutron losses to reactor materials (other than fuel and fertile materials) in typical U^{233}-Th reactors of the aqueous-homogeneous, molten-salt, and gas-cooled types. The materials considered include fission fragments, corrosion products, recombination catalyst, moderator, core vessel, fuel-element cladding, materials associated with coolant channels, and fuel diluent. Expressions were formulated which estimate the loss in breeding ratio due to these neutron poisons in terms of the ratio of poison to fuel absorption cross sections. (AEC Activity 4510)

ISOTOPE PRODUCTION

Radioisotope Production. — Approximately 1.5 curies of C^{14} was separated. The isotopic ratio was about 80%.

Radioisotope Development. — The testing of equipment in the process cells continued at the Fission Products Pilot Plant. Major difficulties encountered were the malfunction of automatic valves in the service lines to process equipment, leakage of a diaphragm in a remote-head pump, leakage from the top cover seal of the centrifuges, and a high corrosion rate in crystal sampler lines which had been plugged with ammonium alum crystals. Maintenance work required for revisions to correct the faults is nearing completion. The installation of Cs^{137} final processing equipment is in progress.

The fabrication and testing of all six model II shielded transfer tanks were completed. Some small voids in the lead shielding were repaired by adding lead sections to the exterior of the containers. Three 500-gal Redox waste shipments were made from Arco to ORNL. Two rail cars were modified for the shipment of three tanks on each car, and one car containing three tanks was dispatched to Arco for filling.

The procedure for separating Ce^{144} from a fission-product mixture, consisting in firing the $Ce_2(C_2O_4)_3$ to CeO_2, pressing pellets of the oxide, and sintering, was tested on 1000 curies of Ce^{144} for the first time. The separation of trivalent rare earths from Ce(IV) was not so sharp as in previous tracer runs. Also it appears to be necessary to use 0.01 M $KMnO_4$ as a holding oxidant in addition to $(NH_4)_2S_2O_8$ to maintain cerium in the higher valence state in the presence of H_2O_2, which is produced by the action of the radiation in the solution.

The efficiency of the ORR caustic scrubber, which is installed in the building ventilation system to remove I^{131} from the exhaust air in an emergency, was found to be 99% by a series of experiments utilizing I^{131} tracer. (AEC Activity 5100)

Stable Isotope Separation. — Calutron separations of both silver and ytterbium isotopes were completed, yielding an estimated 9.8 g of silver and 87.9 g of ytterbium. The enhancement of Yb^{168} (normally 0.14% abundant) reached a value of 365.

The first calutron separation of lutetium isotopes is in progress. The present yield is estimated to be 14 g.

Copper isotopes are being separated to achieve a higher purity Cu^{65} sample than has been produced previously. The current production of Cu^{65} is 28 g; the expected purity is approximately 99.7%.

Charge materials of Cu_2I_2, Cu_2Cl_2, $YbCl_3$, and $LuCl_3$ were fused by high-frequency induction heating before submission to the separations group in kilogram quantities.

Chemical refinement was completed on new lots of antimony and selenium and on one returned lot of copper. New samples of antimony and selenium and returned samples of copper, antimony, gadolinium, and europium were assayed and prepared for inventory.

Preliminary samples of copper, silver, and ytterbium were recovered from graphite collectors, homogenized, and purified to obtain comparative "in-run" data.

Twenty-four conversions to other than inventory form and 20 target preparations were completed.

Stable Isotope Development. — Current separations of copper isotopes are being used to further evaluate new high-purity equipment. A sample of Ag^{109} produced recently under similar conditions showed an eightfold increase in enhancement over the best previously produced sample.

Magnet pole faces for use in an electromagnetic isotope separator having an inhomogeneous (radially decreasing) field were completed. (AEC Activity 5100)

PHYSICS

High Voltage Program. — The new 60-deg reaction particle magnet has been installed on the 5.5-Mv Van de Graaff, and preliminary operational tests have been performed. The magnet is vacuum-tight and exceeds the manufacturer's specifications for uniformity and constancy of field. Tests with elastically scattered protons show that the experimental object and image distances agree very well with the design figures. Measured values of momentum resolution as low as 0.15% have been obtained; this value is somewhat better than the design value.

The neutron absorption cross sections at a neutron energy of 27 kev have been measured for copper, silver, antimony, and gold. Photoneutrons from a spherical antimony-beryllium source were used in spherical-shell transmission measurements, in which the effects of scattering are canceled in the spherical geometry by the use of a detector with a flat response. In these measurements the transmission is related to the total and absorption cross sections of the shell material; previously measured values of the total cross sections are used in the analysis to obtain the absorption cross sections. The absorption cross sections for the elements listed above are as follows: copper, 50 ± 20 mb; silver, 940 ± 70 mb; antimony, 1100 ± 100 mb; gold, 600 ± 100 mb. (AEC Activity 5220)

The 86-Inch Cyclotron: Nuclear Physics. — Excited states of odd-A nuclides in the rare-earth region which decay by electron capture were studied with internal-conversion spectrographs. There are many dozens of transitions in the nuclear spectra of Tm^{161}, Tm^{163}, Tm^{165}, Lu^{167}, and Lu^{169}, with no apparent strong rotational sequence in the ground state. Tentative decay schemes were formed for these previously unknown activities. A new isomeric transition of 211 kev and of E3 multipole order was observed in Ho^{161}. (AEC Activity 5220)

Heavy-Particle Physics. — A conference on Reactions Between Complex Nuclei was held at Gatlinburg, May 5 to 7. Twenty-three papers were presented in a survey of recent American and West European research in all fields of heavy-ion physics. About 80 physicists attended, representing the United States, Canada, England, France, and Sweden. The active Russion research programs were unfortunately not discussed because the three Russian physicists who were invited were unable to attend. A summary of the work at Moscow was received, however, and will be included in the conference proceedings.

A study of the elastic scattering of N^{14} by Be^{9} was completed. Between 32 and 93 deg in the center-of-mass system, the ratio of the cross section to the Rutherford cross section drops from 0.45 to 0.06. Slight indications of structure are seen. (AEC Activity 5220)

Mass Spectrometry and Related Techniques. — An investigation of bimolecular ion-molecule reactions of the type $HCOOH^{+} + HCOOH \rightarrow HCOOH \cdot H^{+} + HCO_2$ was completed. By using isotopically labeled aliphatic acid reactants, it is shown that the hydrogen in the product ion results from rupture of either the C-H bond or the O-H bond, but that rupture of the C-H bond is the preferred process. The high value of 1×10^{-9} cc·molecule^{-1}·sec^{-1} for the bimolecular specific rate constant suggests that the ions react essentially on every collision with the neutral molecules. (AEC Activity 5250)

CHEMISTRY

Mass-233 Studies. — Ion exchange and solvent extraction with diisobutylcarbinol are being considered as possible methods for the removal of Pa^{233} in a Thorex head-end treatment. Slurrying 30 ml of 1/16-in. $Th_3(PO_4)_4$ spheres in 20 ml of 1 M $Th(NO_3)_4$—1 M HNO_3 solution for 10 min removed 30% of the protactinium and of the zirconium and niobium. An ion exchange column containing $Th_3(PO_4)_4$ precipitated interstitially in Dowex 50 resin had a high adsorption capacity for protactinium. Protactinium was not scavenged from the solution by sodium silicate.

The extraction of uranium and thorium by diisobutylcarbinol increased with increasing concentrations of HNO_3, $Al(NO_3)_3$, or $Th(NO_3)_4$ in the feed solution. At 3 M H^{+}, 0.5 M $Al(NO_3)_3$, and 0.2 M $Th(NO_3)_4$, the uranium distribution coefficient was 0.08; it increased to 0.6 as the $Th(NO_3)_4$ concentration

increased to 1.3 M. In a solution containing 0.5 M $Al(NO_3)_3$ and no acid, the thorium distribution coefficient was 10^{-4}; it increased to 3×10^{-2} as the acid increased to 5 M.

Although uranium and thorium are much more soluble in dibutyl butylphosphonate (DBBP) and diamyl amylphosphonate than in tributyl phosphate (TBP), fission products are also much more soluble.

In a countercurrent batch extraction under Thorex extraction column conditions, with 35% DBBP instead of 42.5% TBP as the extractant, decontamination factors for ruthenium, for zirconium and niobium, and for total rare earths were 20, 125, and 550, respectively. These values are lower by a factor of 10 than those with TBP. Higher saturations of the solvent would be difficult to maintain in the pilot plant owing to the difficulty of maintaining exact flow rates of process solutions. A third phase was formed when a 30% DBBP solution was used.

Preliminary experiments to determine the chemical feasibility of the use of boron or rare earths as a homogeneous neutron poison for criticality control showed that the neutron poisons are not separated during head-end treatments but are removed by a 1.5% TBP solvent extraction cycle. (AEC Activity 5310)

Raw Materials Chemistry. — Uranium was extracted from sulfuric acid leach slurries of bomb liner salvage by both the Amex and Dapex processes without the formation of troublesome emulsions. Dapex process data indicated that more than 99% of the uranium can be recovered in four countercurrent stages from a slurry containing 20% solids and having 250 g of SO_4 per liter and a pH of 0.3.

In extractions of Np(IV) and thorium from nitrate solutions by trialkyl amines in both xylene and Amsco 125-82 diluents, single-stage separation factors were 10 to 15 at various acidities and thorium concentrations. The distribution coefficients of Np(IV) were greater than 100 from 8 M HCl, while thorium coefficients were less than 0.01.

The stoichiometry of the stripping by sodium carbonate in the Dapex uranium recovery process was verified in laboratory tests. For complete stripping the base must be supplied in amounts at least equivalent to the free (uncomplexed) acid reagent and to the uranium organophosphate, for example,

$$2\ HR + Na_2CO_3 \longrightarrow 2NaR + CO_2 + H_2O$$

$$UO_2R_2 + 3Na_2CO_3 \longrightarrow 2NaR + Na_4UO_2(CO_3)_3$$

or

$$UO_2R_2 + 6NaHCO_3 \longrightarrow 2NaR + Na_4UO_2(CO_3)_3 + 3CO_2 + 3H_2O$$

where HR is the uncomplexed dialkylphosphoric acid, and UO_2R_2 is the uranyl

dialkylphosphate complex. When carbonate solutions were used at concentrations calculated from the above equations and CO_2 gas was purged from the system, strip solutions were prepared that were essentially water solutions of sodium uranyl tricarbonate, $Na_4UO_2(CO_3)_3$. With less base, uranium was reextracted into the organic phase in amounts corresponding to the destruction of $Na_4UO_2(CO_3)_3$ by free acid according to the equation

$$6HR + Na_4UO_2(CO_3)_3 \longrightarrow 4NaR + UO_2R_2 + 3CO_2 + 3H_2O.$$

(AEC Activity 5310)

Chemical Engineering Research. — The interfacial area in an all-quartz pulsed column with a fluorocarbon discontinuous phase was measured by the $F(\alpha,n)$ reaction; the area increased linearly with the throughput and holdup of the discontinuous phase; pulse amplitude had little effect. (AEC Activity 5310)

Equipment Decontamination Studies. — The chief problem in the decontamination of stainless steels with respect to mixed fission products is the removal of niobium. Stainless steel was contaminated for test purposes by heating in an aqueous acidic salt solution containing Zr^{95}-Nb^{95} tracer (0.2 mc/ml) at 150°C for 18 hr. The resulting activity of about 7 mr/hr on 1.5 in.^{2} of steel was strongly bound and was not removed by boiling for several minutes with 5 N HNO_3. The contamination was lowered by a factor of at least 10 by isotopic dilution of the Nb^{95} in the contaminating solution with about 0.4 g/liter of nonirradiated niobium as the peroxyniobate. As a decontaminating reagent for Nb^{95} and Zr^{95}, 3% H_2O_2 in 5 M HNO_3 was more effective, cheaper, and more stable than alkaline tartrate-peroxide. Studies on its possible corrosiveness are still incomplete. Stainless steel coupons that had been contaminated by isotopically diluted Zr^{95}-Nb^{95} were decontaminated to (0.01 ± 0.01) mr/hr by gentle boiling for 5 min in acidic peroxide solution. (AEC Activity 5310)

Extraction Reagent Performance. — Solutions of tributyl phosphate (TBP) and nitric acid in Amsco were irradiated to 450 whr/liter. The extraction of uranium by these solutions decreased, nonlinearly, as the TBP concentration increased from 150 to 600 g/liter in Amsco. At 600 g of TBP per liter, the extraction efficiency of the irradiated solution was only 25% of that of the unirradiated control solution. The emulsification tendencies of these same irradiated solutions were proportional to the TBP concentration in the range 150 to 450 g/liter. Doses of 450 whr/liter increased the degree of emulsification, as compared with unirradiated control samples, by factors of 3 to 4. The solubility of thorium dibutyl phosphate in TBP-Amsco solutions was less than 0.04 mg/ml. (AEC Activity 5310)

Feed Materials Processing. — In studies on the direct reduction of UF_6 to uranium metal, 86% of UF_6 vapor carried in argon at 25 to 50°C was reduced to UHg_4 by a 133 to 150% stoichiometric excess of 6 M lithium amalgam. The by-products of the reduction and the bulk of the spent amalgam were separated

from the uranium quasi amalgam by centrifugation. The lithium amalgam was prepared by electrolysis of 2 M LiOH at room temperature. The 3 M lithium amalgam that formed at first later separated into a 0.9 M lithium amalgam solution and a 6 M lithium amalgam sludge that floated and was skimmed from the top. (AEC Activity 5310)

Ion Exchange Technology. The distribution coefficients for U(VI) and U(IV) between Dowex 1 resin and sulfuric acid solutions increased approximately as the square of the reciprocal of the sulfate concentration.

An equilibrium constant of 23.1 was calculated for the two-phase system of anion exchange resin IRA-400 and sulfate-bisulfate solution, with data taken from two different literature sources. (AEC Activity 5310)

Organic Chemistry. In a continuation of studies of the mechanism of the decarbonylation of carboxylic acids by sulfuric acid, the C^{14} isotope fractionation has been studied for the decarbonylation of triphenylacetic-α-C^{14} acid. At 25°C, $k_{12}/k_{14} \leq 1.01$, indicating that cleavage of the carbon-to-carbon bond to the carboxyl group has little to do with the reaction rate. The deuterium isotope effect reported earlier for the decarbonylation of DCOOH showed that cleavage of the carbon-to-hydrogen bond had little influence on the reaction rate.

Since the result with triphenylacetic acid may be related to the formation of the highly resonance-stabilized triphenylmethyl carbonium ion intermediate, a third isotope fractionation study, namely, of the decarbonylation of benzoylformic-α-C^{14} acid in concentrated sulfuric acid, was carried out. The corresponding intermediate ion in the latter case is believed to be the benzoyl carbonium ion, which has fewer possible resonance forms than the triphenylmethyl ion. At 25°C the decarbonylation of benzoylformic-α-C^{14} acid gave $k_{12}/k_{14} = 1.036$, about half the expected maximum isotope effect for carbon-to-carbon bond cleavage. The mechanism of decarbonylation of benzoylformic acid may involve a concerted rate step in which both carbon-to-carbon bond cleavage and carbon-to-oxygen bond cleavage play significant parts.

Cyanogen was shown to undergo ortho addition when allowed to react with various benzyl-type Grignard reagents.

The large isotope effects (reported by other investigators) in the rearrangement of labeled pinacols were reinvestigated and found to be in error. (AEC Activity 5330)

Radiation Chemistry. In the sequence of events following the absorption of high-energy radiation in liquid water, H_2 and H_2O_2 are produced, presumably by the combination of radicals as they diffuse away from sites of formation along the tracks of charged particles. Information, both on the

nature of the processes leading to these products and on the initial spatial distribution of the intermediates, can be gained by studying the effects of added scavengers of radicals. In general, solutes that react with H atoms lower the H_2 yield, and those that react with OH radicals lower the H_2O_2 yield. For irradiation by Co^{60} gamma rays, the decrease in yield is, in a number of cases, proportional to the cube root of the scavenger concentration in the range up to ~0.1 M. This dependence is consistent with the radical diffusion model developed by Magee and others. The yields of hydrogen from degassed solutions of $NaNO_3$ (pH 5.1 to 5.8) and HNO_3 in the range of concentration up to 1.0 M were identical and could be expressed by $G_{H_2} = 0.46 - 0.46a^{1/3}$. The results indicate dependence on the activity of the solute rather than on the molarity. This was confirmed by changing the ionic strength by adding NaCl at concentrations of 1.0, 3.0, and 5.0 M to 0.1 M $NaNO_3$ solution. In disagreement with published results, the data indicate no effect of acidity on the "molecular" H_2 yield or on the action of NO_3^- in lowering the H_2 yield. Also, H_2SO_4 (0.01 to 0.4 M) and Na_2SO_4 (up to 1.8 M) had no effect on the H_2 yield; hence sulfate is not reactive with H atoms, and if appreciable excitation of the solute occurred, then this energy was effective in producing H_2. The yield of "molecular" H_2O_2 in 0.4 M sulfuric acid solution is the same for both H_2O and D_2O, and KBr lowers this yield equally for both systems: $G_{H_2O_2} = G_{D_2O_2} = 0.81 - 1.65(KBr)^{1/3}$. The yields of H_2, H, and OH are 0.45, 3.68, and 2.96, as compared with the values 0.38, 3.99, and 3.14 for D_2, D, and OD. (AEC Activity 5330)

Molten Salt Thermodynamics. — For comparison with a similar study of the AgCl-NaCl system, free-energy data and related thermodynamic quantities have been obtained for solid and liquid solutions of AgCl and LiCl between 300 and 900°C from galvanic cells of the type $Ag/AgCl,LiCl/Cl_2$. In the liquid solutions, greater positive deviations from Raoult's law were found with LiCl than with NaCl. In the solid solutions, the change of the activity of AgCl with composition was not monotonic; the shape of the activity curves suggests that the concentration range in which the solid solutions are metastable with respect to separation into two phases is greater in the case of the AgCl-LiCl system than it is in the case of the AgCl-NaCl system. (AEC Activity 5330)

Analytical Chemistry Research. — A rapid, yet precise, method was developed for the determination of thorium in monazite sands and in solutions which are derived from the leaching of those sands with sulfuric acid. After the addition of nitric acid and sodium nitrate to overcome the complexing effects of phosphate and sulfate on thorium which prevent its extraction, the thorium is extracted by a single equilibration with a 0.1 M solution of tri-n-octylphosphine oxide (TOPO) in cyclohexane. Under these conditions, the rare-earth elements are not extractable. Thorium is re-extracted from the cyclohexane solution with 0.3 M H_2SO_4, after which it is estimated by the spectrophotometric Thorin method. In comparison with other methods for determining thorium, this procedure takes about 1/10 the time and introduces no loss in precision (1% relative).

In the continuation of the studies on the uses of tri-n-octylphosphine oxide as an analytical reagent in the separation of metals by solvent extraction, work was initiated to ascertain the extraction characteristics of tin. Extraction coefficients of the order of 1000 were obtained for tin(IV) in 1 to 5 M HCl solutions. If the extraction is carried out from more concentrated acid solutions, the extraction coefficient decreases sharply to about 10 from 10 M HCl. Preliminary data indicate that the extracted species contains two moles of TOPO per mole of tin.

METALLURGY

Fundamental Physicometallurgical Research. — In order that the stability of initial orientations in polycrystalline metals might be investigated, an aluminum rod containing an orientation of ⟨118⟩ was cold-swaged to give reductions in area of 60, 80, 90, and 96%. The axis distribution charts showed that approximately 40% of the volume quickly moved directly toward a ⟨111⟩ orientation and that the remaining 60% moved more slowly to a ⟨001⟩ orientation. Traces of the initial ⟨118⟩ texture persisted up to a reduction in area of 90%.

The specific heats of several zirconium-indium alloys have been measured in the liquid-helium temperature range. The electronic part of the specific heat increases linearly, and the Debye temperature decreases in a parabolic fashion with indium additions. The changes were 13 and 4%, respectively, for the largest addition of 8% indium.

In the zirconium-cadmium system, vapor pressure investigations of the beta phase have shown that heat is released upon mixing these elements and that the entropy of formation is less than the ideal value. Apparently either a deviation from random mixing or a significant contribution to the specific heat from alloying occurs.

Zirconium phase diagram studies have shown that the peritectic reaction in zirconium-lead occurs at 880 ± 10°C and that the solubility limit of cadmium in alpha zirconium is 11.5 at. % Cd at 880°C. (AEC Activity 5420)

Microstresses in Metal Crystals. — Experiments to measure the imperfections in very thin surface oxides on single crystals by a diffraction technique continue. Diffraction maxima from films approximately 190 and 540 Å thick and grown on a 110 face of copper were measured, and work continues on the interpretation of their shapes in terms of the imperfections present in the films.

To study the effect of reactor radiation on the state of order in Cu_3Au, a single crystal of this alloy has been prepared from which specimens of convenient orientations have been cut.

Work was completed on an extension of the diffraction theory for the atomic size effect in substitutional disordered alloys.

The crystal scintillation detector for the diffractometer was completed and installed, and is now in use. (AEC Activity 5420)

Fundamental Investigations of Radiation Damage in Solids. — The effect of irradiation by Co^{60} gamma rays on the concentrations of carriers in high-purity n- and p-type germanium single crystals was studied further. Previous results indicated that a temperature-sensitive acceptor level was introduced 0.2 ev below the conduction band. This was tentatively identified as the first ionization energy of the interstitial atom produced by irradiation. The results of annealing at a moderate temperature (100°C) suggested a preferential migration of such interstitials, since the state was almost totally removed by such annealing.

Continued irradiation of n-type material causes conversion to p-type, with the introduction of an acceptor level located 0.24 ev above the valence band. This level is presumably associated with the first ionization energy of a vacancy produced by irradiation. Annealing at a moderate tmperature (100°C) has no effect that would suggest either preferential migration of vacancies or extensive recombination of interstitial-vacancy pairs.

No evidence of low-lying acceptor levels has been found for material irradiated by Co^{60} gamma rays. This is in contrast to the results obtained with fast neutrons, where several shallow acceptor levels or hole traps were observed. (AEC Activity 5430)

PROGRAM 6000 — BIOLOGY AND MEDICINE

BIOPHYSICS

Radiation Dosimetry Programs. — The method for measuring stopping power of matter for low-energy electrons which was reported previously (ORNL-2393) has been changed in order to obtain increased sensitivity. The temperature of the foil is now measured directly with an array of six thermocouples in series electrically and spaced around the periphery of the foil. The drift rate of the galvanometer connected in series with these thermocouples is directly proportional to the power expended in the foil by the passage of an electron beam, with a calibration constant of 40 $cm \cdot min^{-1} \cdot mw^{-1}$. This constant is obtained by reducing the beam energy until complete absorption in the foil is accomplished, thus delivering a known amount of power to the foil for which the corresponding galvanometer drift rate may be noted. An identical independent set of thermocouples similarly positioned provides a means of cooling the foil by Peltier cooling before an experimental run, so that the rate of drift

may be obtained as the foil passes through room temperature, thus eliminating the need for corrections due to heat transfer to the surroundings.

For an aluminum foil 108 $\mu g/cm^2$ in thickness, the measured stopping power varies from 3.5 $kev \cdot mg^{-1} \cdot cm^2$ at 100 kev to 18 $kev \cdot mg^{-1} \cdot cm^2$ at 10 kev, in good agreement with the Bethe theory. The stopping power at the latter energy includes a 24% correction to foil thickness obtained from a modified Yang theory, in order to take into account the increase of the electron path length over the actual foil thickness due to multiple scattering. (AEC Activity 6140)

Waste Disposal Research: Soil Disposal of Intermediate-Level Radioactive Wastes. — From June 1952 through December 1957, 8.6 million gal of waste containing 112,000 curies of beta activity was released to the pit system. The radioactivity released includes 68,000 curies of Cs^{137}, 19,000 curies of Ru^{106}, and lesser amounts of Sr^{89}, Sr^{90}, Co^{60}, Sb^{125}, I^{131}, Pa^{233}, and Ru^{103}.

While the general pattern of waste movement from the pits has been established, the movement of individual radionuclides has not been defined in as much detail as desired. The movement of ground water and any mobile waste materials present is restricted to the north of the pits and cannot extend beyond the streams east and west of the pits. To the south the streams drain into White Oak Creek. The liquid escaping from the pits is monitored in its travel at numerous wells, at weirs located on the streams, and before discharge from White Oak Creek to the Clinch River. Thus the material escaping from the pits cannot inadvertently become a hazard even if the details of its movement are unknown.

To date the two most hazardous isotopes, Sr^{90} and Cs^{137}, have not been detected in waste escaping from the pits. Ruthenium-106 is the only isotope that has been identified in important amounts in any of the observation wells, seeps, and streams. Laboratory and field results indicate that a continuing removal of ruthenium occurs as long as the waste remains in contact with the shale. The operation of the waste-pit system from 1952 through 1957 has been responsible for the release of about 150 curies of Ru^{106} from White Oak Creek to the Clinch River. (AEC Activity 6440)

Waste Disposal Research: Process Waste Water Treatment Plant. — A total of 58.55 million gal of process waste was treated during the first six months of plant operation. This amounted to 43% of the total process waste, 57% being bypassed and discharged to White Oak Creek without treatment. Removal of the important radiostrontium ranged from 66.4 to 87.4% for monthly periods, and was as high as 91.6% for individual periods of a week. The total rare earths are removed as efficiently as the strontium. A total of 16.93 curies of strontium and 14.73 curies of total rare earths was removed. The activity was associated with 208,000 gal of sludge, which was transferred to the waste disposal pit system. The amount of calcium remaining in the plant effluent is indicative of the efficiency of removal of strontium and the total rare earths. Therefore high water-softening efficiency is a requisite. The

presence of Versene in the waste is detrimental to efficient removal of calcium. Low temperatures also restrict the removal of calcium. The addition of 30 ppm of a local clay to the waste increased the removal of radiocesium from about 16 to 55%. (AEC Activity 6440)

PROGRAM 8000 — WORK FOR OTHERS

Professional Health Physics Training Program. — Twenty-seven AEC Fellows, one civilian with the Army, and one Air Force officer completed their academic training at Vanderbilt University at the end of May. Four of the AEC Fellows were assigned to the Idaho Falls Reactor Testing Site for their training in applied health physics. The remainder of the group reported to Oak Ridge on June 4. One Air Force officer, who had taken his academic training at the University of Rochester, was transferred to the Oak Ridge National Laboratory for his training in applied health physics.

During May, one member of the Education and Training staff spent three weeks on the work of the Publications Committee of the International Commission on Radiological Protection. (AEC Activity 8780)

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VISITORS

FOREIGN VISITORS:

Bariand, A., French AEC
Bjornerstedt, R. G., United National Committee on Radiation (Swedish Citizen)
Buckingham, R. A., University of London, England
Chester, P. F., Westinghouse (Citizen of Great Britain)
Clarke, J. C., United Kingdom AEA
de Waegh, F. W., S.E.E.N., Belgium
D'Orval, C. M. C., French AEC
Egelstaff, P. A., United Kingdom AEA
Faires, R. A., AERE, Harwell, England
Galbraith, W., AERE, Harwell, England
Gorter, E. W., Philips Research Labs., (Citizen of The Netherlands)
Haeszner, F. W., Max Planck Institute, Mainz, Germany
Hjort, P. F., University of Oslo, Norway
Hudswell, F., AERE, Harwell, England
Isobe, T., Furakawa Electric Company, Ltd., Japan
King, N. M., AERE, Harwell, England
Love, H. M., Queen's University, Ontario, Canada
Manil, P. A., C.E.N., Belgium
Miyata, K., Sumitomo Shoji, Inc., Japan
Miyazaki, I., Japanese Government, Japan
Miyoshi, E., Sumitomo Metal Industries, Japan
Nagahata, Y., Hitachi Shipbuilding Co., Osaka, Japan
Niomura, N., Hitachi Shipbuilding Co., Osaka, Japan
Patterson, G. T. G., United Kingdom Atomic Energy Authority
Pedretti, E., Pennsylvania State University (Citizen of Italy)
Petit, J. F., French AEC
Pryce, M. H. L., University of Bristol, England
Roth, E., Comissariat A L'Energie Atomique, France
Sabine, T. M., Australian AEC
Saijo, I., Mitsubishi - Metal Mining Co., Japan
Sawada, M., Osaka University, Japan
Schlogl, R., Max Planck Institute, Göttingen, Germany
Sweetman, D. R., United Kingdom A.E.A.
Takahashi, S., Government of Ibaraki - Prefecture (Japan)
Takeda, S., Hosei University, Japan
Teng, L. C., Argonne National Laboratory (Citizen of China)
Tolun, R., Laboratory, Mining and Exploration Institute, Turkey
Von Gunten, H. R., Reactor, Ltd., Switzerland
Watanabe, S., University of Tokyo, Japan
Ward, J. C., University of Maryland (Citizen of Gr. Britain)
Woldringh, S. H., Reactor Center, Netherlands
Yamaguichi, K., Atomic Energy Bureau of Japan

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145. H. E. Seagren
146. E. D. Shipley
147. A. Simon
148. O. Sisman
149. J. R. Sites
150. M. J. Skinner
151. A. H. Snell
152. R. W. Stoughton
153. H. Stringfield
154. E. G. Struxness
155. W. H Sullivan
156. C. D. Susano
157. J. A. Swartout
158. E. H. Taylor
159. B. S. Weaver
160. A. M. Weinberg
161. J. C. White
162. C. E. Winters
163. H. P. Yockey
164. W. Zobel
165-166. Biology Library
167-168. Central Research Library
169. Health Physics Library
170. ORNL—Y-12 Technical Library Document Reference Section
171. R.E.E.D. Library
172-174. Laboratory Records Department
175. Laboratory Records ORNL, R.C.

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