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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

AUGUST 1963

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

AUGUST 1963

This Status and Progress Report summarizes the unclassified portion of the Laboratory's work. Some of the topics are included every month, but the majority are reported on a bimonthly schedule.

REACTOR DEVELOPMENT

GAS-COOLED REACTOR PROGRAM

Procurement of the EGCR Fuel Assemblies. – Fabrication of the first core loading and spare fuel assemblies was completed. All assemblies were accepted by ORNL and are now stored at ORGDP.

AVR Physics. – The nuclear characteristics of additional AVR reactor (German pebble-bed reactor) fuel loadings were investigated to determine whether plutonium produced from partially enriched uranium would have a beneficial effect on the shutdown margin. Three compositions were studied: (1) thorium and fully enriched (90%) uranium with a thorium-to-total-uranium ratio of 7; (2) thorium and 30%-enriched uranium with a thorium-to-uranium ratio of 2; and (3) 12%-enriched uranium with no thorium. In all three cases the fuel spheres contained 5 wt % heavy metal with 1/3 of the spheres fueled, the fuel was distributed in two fueled regions with the fuel-sphere fractions adjusted to given equal average outlet gas temperatures, and boron was used as a control poison. The thorium-to-uranium ratios of 7 and 2 were selected because they would give a small or negative reactivity change upon the addition of steam to the core. For the case with no thorium, however, there would be a significant reactivity increase upon steam addition, but the case was studied to determine the effect of larger amounts of plutonium on other characteristics. The lifetimes up to complete boron removal were 187, 81, and 193 days, respectively, for the three cases. At those times, the shutdown temperatures after xenon decay with three rods inserted were 157, 150, and 184°C from an operating temperature of 627°C.

ORR Loop No. 1 Irradiation Tests. – Operation of ORR loop No. 1 with a stainless-steel-clad UO_2 fuel element, of the EGCR type, that had been deliberately made defective by drilling a 1/16-in.-diam hole through the cladding was completed successfully. The equilibrium fission gas concentration in the loop of approximately 1 curie, achieved at a cladding

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surface temperature of 1500°F and a central fuel temperature of 2100°F, was approximately that expected for fuel which had a D' (release rate parameter) of $\sim 10^{-11}$ sec^{-1}. Contamination levels in the loop following evacuation and refilling with a fresh helium charge were not significantly higher than previous levels. After the fuel had been removed from the loop, both compressor housings were opened and the rotating parts were changed without difficulty. These findings present an encouraging prospect for maintenance of EGCR-type reactors in the event of fuel element failures.

ORR Loop No. 2 Irradiation Tests. — The first experimental assembly containing four 1.5-in.-diam spheres consisting of coated uranium carbide particles in a graphite matrix was irradiated throughout one ORR cycle of operation. The average heat generation for the spheres was ~ 30 w/cm^3 during operation at an inlet gas temperature of ~1060°F. Release of fission gas from the fuel was low throughout the run, with an estimated concentration of krypton and xenon activity of less than 25 $\mu\mu c/cm^3$.

A new experimental assembly containing three 6-cm-diam AVR-type fuel spheres consisting of coated-particle fuel in a graphite matrix with a 1/4-in. unfueled shell was assembled and installed in the loop for operation during the next cycle. The assembly was altered to provide enough bypass helium flow to enable achievement of the desired fuel inlet gas temperature of 1200°F while maintaining the necessary operating conditions in the loop.

Capsule Irradiations in the ORR Poolside Facility. — Irradiation of three sweep capsules loaded with 1-1/2-in.-diam spheres containing pyrolytic-carbon-coated uranium carbide particles in a graphite matrix was completed. The ratio of fission-gas-release rate to birth rate for Kr^{88} for these capsules was $\sim 1.5 \times 10^{-5}$ to 2.5×10^{-5} near the end of irradiation at an estimated burnup of 5 at. % total uranium. Three new capsules containing 6-cm-diam fueled spheres of the AVR type were prepared and installed for irradiation. Each capsule contains three spheres, one in a sweep compartment for continuous measurement of fission gas release and the other two in a single static compartment.

Eight-Ball Experiment. — Irradiation of the second experimental assembly containing eight 1-1/2-in.-diam spheres fueled with coated particles of uranium carbide in a graphite matrix was completed, and a third assembly was installed. The spheres in the second assembly operated at a power density of ~ 40 w/cm^3 and a surface temperature of ~1100°F.

Instantaneous Fission Gas Release from Pyrolytic-Carbon-Coated Uranium Carbide Particles. — Capsule B9-11, containing pyrolytic-carbon-coated $(U,Th)C_2$ particles from batch NCC-208-2, was irradiated at 2050°F to 14.7 at. % burnup of the heavy metal. The uranium-to-thorium ratio was 1.5. The fractional gas release rates were lower than those for other particles irradiated in this series of experiments, and postirradiation examination indicated that none of the coatings were cracked or broken.

Capsule B9-12, containing particles from the same batch as capsule B9-11, was irradiated for four days at 2150°F. The gas release was low, but when the temperature was increased to 2300°F after 2.4 at. % burnup of the heavy metal, the fission gas release became excessive.

Capsule B9-13, containing UC_2 particles with triplex pyrolytic carbon coatings from batch GA-308, was irradiated to 12 at. % burnup of the uranium at 2400°F. The average fractional fission gas release was low, and no increase in fission gas release was observed during the test. After 12 at. % burnup of the uranium, the neutron flux was remeasured; during the measurement the specimen temperature was increased from 2300 to 2400°F. At the time of the increase in temperature, a burst of gas indicated that one or more coatings had ruptured. After the flux measurement had been completed, the specimen was irradiated for two days at 2300°F; then the temperature was increased to 2400°F to see whether an increase in operating temperature would damage the particles. After 130 min at 2400°F, it appeared that coatings began to rupture, and after 8 hr the released activity had increased by a factor of 1000. The irradiation was then terminated.

Fission-Product Deposition Studies. — A computer code designed to illustrate an analytical model for the rate of deposition of fission products from a gas stream onto the surface of a conduit in isothermal and nonisothermal regions was completed. The code is designed to handle fully developed laminar and turbulent flow. The output gives the rate of deposition as a function of distance along a conduit.

Fueled-Graphite Handling Tests. — Fueled-graphite spheres from four vendors gained weight during abrasion tests. In an attempt to understand this, the spheres were outgassed in vacuum at 1000°C for 5 hr, during which they lost weight. Then they were exposed to laboratory air for seven days, during which they again gained weight; the spheres which had the highest prior weight gain displayed the greatest weight gain during the seven-day period in air. This confirms the hypothesis that the weight gains were due to adsorption of atmospheric gases during the test period.

Steam-Graphite Reaction Tests. — A representative EGCR graphite sleeve is being tested to determine the hydrogen production to be expected as a result of the passage of steam over the graphite sleeves in the event of an internal rupture of a steam generator. In the test system the noncondensable gases produced from both the water-gas reaction and the outgassing of the graphite are trapped in the test-loop condenser and measured. Based on the average gas-production rate and mass spectrometer analyses of the gas produced, calculations are made of the production rates of hydrogen, CO_2, CO, and CH_4. In the current test, the graphite sleeve was operated for ~460 hr at an average inlet temperature of 1390°F and a system steam pressure of 150 psig. During this period the specimen lost 350 g, or 0.53 g of graphite per square foot per hour, which is substantially less than the loss factor assumed in analytical calculations associated with the EGCR hazards review.

ARMY POWER REACTORS

Water Chemistry. — In a continuing study of possible methods for decontamination of pressurized-water-reactor coolant at elevated temperatures (ORNL-3477, p 4), a sampling system was assembled for passing filtered water from the ORR pressurized-water in-pile loop over various high-temperature adsorbents to test their efficiencies for removing soluble radioactive material circulating in loop water. The first out-of-pile tests of the system showed that Fe_2O_3 was precipitated when a 10^{-6} M Fe^{2+} solution containing approximately 1 ppm O_2 was heated to 260°C, even though the solution had been saturated at room temperature with H_2 at 1 atm.

No formation of Fe_2O_3 was observed in a second test, in which the O_2 concentration was reduced to approximately 0.01 ppm. A sample of Fe_3O_4, which was being tested as an adsorbent at 260°C, retained most of the Fe^{2+}. The average surface area associated with each adsorbed iron atom was about 7 A^2, an indication of extensive adsorption.

The solubility of Fe_3O_4 in water at the operating temperature of pressurized-water reactors is being investigated because of its importance in understanding the transport of corrosion products and associated activity through these systems. This solubility is expected to be strongly dependent on the pH of the water, according to the reaction

$$3Fe_3O_4 + H_2 + 6H^+ = 3Fe^{2+} + 4H_2O \ .$$

Tests with pure water have given erratic results. Preliminary results with 10^{-5} M HCl indicate that at 260°C 1 to 3 ppb Fe_3O_4 (2 to 6 × 10^{-8} M) dissolves in H_2O having a room-temperature pH of 5.5.

REACTOR FUELS AND MATERIALS

Radiation Effects in Structural Materials. — The effect of neutron irradiation on the precipitation of nitrogen in iron was investigated by making internal-friction measurements during irradiation of specimens in the ORNL Graphite Reactor. The specimens were vibrated in flexure at 35 cps so that the peak of the internal-friction curve due to the motion of nitrogen atoms (Snoek peak) was at 63°C.

In the first set of measurements, made in Hole 17 on a specimen containing 0.022 wt % nitrogen, it was not possible to observe the rate of decay of the nitrogen peak. However, the internal-friction measurements made after most of the nitrogen in solution had precipitated showed some features not found in unirradiated specimens. The nitrogen peak leveled off at a value much higher than that observed in out-of-pile measurements, but after the reactor was shut down the height of the peak decreased rapidly. In addition, the measurements indicated a rise in internal friction at temperatures below the nitrogen peak.

A second set of measurements was made in an attempt to observe the low-temperature internal-friction peak indicated by the earlier measurements. In this experiment, carried out in Hole 50 of the Graphite Reactor, where a cooling system permitted measurements to be made at temperatures down to −120°C, a specimen containing 0.015 wt % nitrogen was used. The unusual effects observed earlier were much smaller in this set of measurements. The reduction in these effects might be explained by the lower nitrogen content of the second specimen and the lower neutron flux in Hole 50.

In the second series of measurements the rate of decay of the nitrogen peaks was observed at 63°C after irradiation at −120°C. It was possible to put the nitrogen back in solution after an aging experiment by heating the specimen to 400°C in the reactor and cooling rapidly. The precipitation of nitrogen after an irradiation of 1 day at −120°C (integrated flux, 4×10^{15} neutrons/cm^2, $E > 0.6$ Mev) occurred two to three times as fast as it did in an unirradiated specimen. However, the nitrogen did not precipitate any faster after a 23-day irradiation at −120°C than it did after the 1-day irradiation, thus indicating a saturation in the irradiation effect.

Behavior of High-Temperature Materials Under Irradiation: Irradiation Effects on BeO. — The 1/4-in.- and the 1/2-in.-diam capsules of experimental assembly ORNL-41-10 (100°C) have been disassembled and the pellets recovered. The 1/2-in.-diam capsules were subjected to a gas-sampling operation to determine the presence of any free helium in the capsule void spaces. Trace amounts of helium were recovered; the amounts varied in relation to the neutron dose accumulated by each capsule. It seems evident from these results that less than 0.1% of the helium, generated by the predominating (n,2n) reaction ($Be^9 + n \rightarrow Be^8 + 2n$, $Be^8 \rightarrow 2He^4$), escapes from the small compacts irradiated at 100°C.

High-temperature assembly ORNL-41-8 has been shipped from Idaho to ORNL and is ready for disassembly in the hot cells. It contains BeO compacts and single crystals irradiated at 1100 and 650°C to an estimated dose of 8×10^{21} nvt (fast), the highest dose yet achieved in this series of tests.

A new low-temperature (200°C) experimental assembly containing MgO, Al_2O_3, BeO, and pyrolytic graphite was completed and is now undergoing irradiation in the ETR. Components and specimens for the high-temperature (800 and 1100°C) counterpart experiment were fabricated.

FUNDAMENTAL HEAT TRANSFER AND FLUID DYNAMICS

Magnetohydrodynamic Studies of Vortex Flow. — Consideration of the unique fluid-mechanical properties of vortex flow stimulated interest in the application of such flow to advanced energy-conversion systems that require operation with a high ratio of tangential to radial velocity. In a device of the type envisioned, energy losses by viscous dissipation through turbulence must be minimized, so that the energy required to sustain the vortex will be small compared with the energy output.

Attempts to reduce turbulence in vortex flow by purely hydrodynamic means have met with limited success. An alternative method that can, in principle, produce a significant stabilizing influence over the entire flow field involves interaction between the velocity field in an electrically conducting medium and a suitably oriented magnetic field. Since, in the energy-conversion applications, operation will be at a temperature high enough to sustain ionization in the gas, the possibility of magnetohydrodynamic stabilization is being considered. A uniform axial magnetic field is, in principle, capable of completely stabilizing inviscid, perfectly conducting, pure vortex flow (1/r velocity profile) against small perturbations without altering the mean flow.

Analytical studies of magnetic stabilization of vortex motion based on a dissipative-flow assumption (viscous fluid with finite electrical conductivity) were conducted, and a class of exact, stationary solutions characteristic of incompressible, magnetohydrodynamic flow with cylindrical symmetry under the action of an externally applied magnetic field in the direction of the axis of symmetry was obtained. Dimensionless forms of the pertinent equations characterizing the stabilization of pure, dissipative vortex flow were also developed. The similarity parameters included the peripheral tangential Reynolds modulus, peripheral magnetic Reynolds modulus, peripheral Alfvén modulus, and the magnetic interaction parameter. Another dependent parameter, the peripheral Hartmann modulus, is also believed to be important.

An exploratory experimental study was initiated with saturated aqueous NH_4Cl as the conducting fluid. Initial experiments demonstrated a significant magnetohydrodynamic stabilization effect for a 1.1-in.-ID tangential jet-driven vortex tube oriented in an axial magnetic field of 62 kilogauss generated by the large-volume test magnet in the ORNL Magnet Laboratory.

Reynolds Stress Measurements. — The experimental Reynolds stress data obtained thus far indicate that turbulence intensities in water may be much less than expected from airflow data taken at the same Reynolds number. Further, the radial variation of turbulence intensities in water flowing in a pipe was greater than expected from airflow data.

The apparatus used in these studies was recently modified because of an unexpectedly high noise level in the output signal of the hot-wire anemometer. Originally, each component of the apparatus was checked independently for noise and found to have sufficiently low levels. However, the power supply noise did not attenuate as expected, and, in fact, contributed from 10 to 40% of the output signal. This was corrected.

The experimental method was modified to obtain the degrees of freedom needed to yield a determinate set of equations. The mean square fluctuating voltage of the wire must be measured to obtain the data from which the components of the turbulent stress tensor are calculated. Difficulty arose in practice because the wire orientation could not be changed freely with two degrees of freedom. The wire was mounted on a support that could only be rotated and thus had only one degree of freedom. This was not sufficient to yield a determinate set of equations; therefore it was necessary to place the wire support in the pipe at two different angles; however, if one of the angles was perpendicular to the pipe axis, the equations were still indeterminate. This fact was well hidden and was discovered only recently.

POWER REACTOR FUEL PROCESSING

Chemical Analysis of Advanced Reactor Fuels. — Studies on the use of a 0.75 M NaF solution at a pH of 6.4 to 6.9 as the medium for the controlled-potential coulometric titration of uranium were continued. Several synthetic reactor fuels were analyzed for uranium in this electrolyte. The average results of the analyses of six portions of each fuel solution are shown in the following table:

Reactor Fuel Type	U(VI) Taken (mg)	U(VI) Found (mg)	Bias (mg)	Standard Deviation (mg)
Consumer Public Power (U-Mo)	6.67	6.68	0.01	0.033
Fermi (U-Mo)	6.67	6.73	0.06	0.048
Foreign Research (U-Al-Si)	6.05	6.01	−0.04	0.079
N.S. "Savannah" (UO_2)	6.67	6.67	0.00	0.077
Molten-Salt Reactor ($LiF-BeF_2-ZrF_4-UF_4$)	6.39	6.37	−0.02	0.102
Av	6.49	6.50	0.01	0.06

Chloride Volatility Corrosion Studies. — Welded specimens of "A" nickel and Haynes alloy No. 25 corroded at 27.3 and 40.4 mils/month, respectively, when exposed to a cycle consisting of 5 hr in Cl_2-CCl_4 at 500°C followed by 5 hr in oxygen at 1000°C. "A" nickel, Nichrome V, and Haynes alloy No. 25 corroded at respective rates of 1.2 mils/month (19 cycles), 2.0 mils/month (18 cycles), and 29.6 mils/month (7 cycles) in cyclic tests consisting of oxidation of wet graphite fuel in oxygen at 800°C for 5 hr, followed by chlorination in chlorine at 500°C for 5 hr.

Hastelloy B corroded at rates of 0.5 and 1.5 mils/month during exposure in vapor and solution phases, respectively, of boiling 3 M $ZrOCl_2$—3 M HCl for 840 hr.

Hydrolysis of Uranium Carbide. — The general study of the hydrolysis of carbides in acid and basic aqueous solutions was continued. The volume of gaseous products obtained from the reaction of uranium monocarbide with sodium at various concentrations (of sodium) increased from 103 ml/g (STP) with 2.0 M NaOH to 115 ml/g with 18.6 M NaOH. The gaseous products from 2, 4, and 6 M NaOH consisted of about 26 vol % H_2 and 72 vol % CH_4. With 8.9 M NaOH, the H_2 increased to 32 vol %, while the CH_4 decreased to 65 vol %. With 12.2 and 18.6 M NaOH, the gaseous products consisted of

35 vol % H_2 and 61 vol % CH_4. In contrast, the gas obtained by the reaction of uranium carbide with water contained 13 vol % H_2 and 84 vol % CH_4. About 92% of the carbon originally present in the uranium monocarbide was found in the gas phase and 0.5% in the sodium hydroxide-insoluble solids. The time for 95% of the carbide to react varied from 2.5 hr with water to 6 hr with 2 M NaOH and 41 hr with 18.6 M NaOH.

THORIUM UTILIZATION PROGRAM

Thorium-Uranium Fuel Cycle Development Facility. — The Thorium-Uranium Fuel Cycle Development Facility (TUFCDF) is to afford adequate shielded space for development of processes suited to remote processing and fabrication of Th-U^{233} fuel assemblies of different types. Sufficient shielding (5-1/2 ft of normal concrete) is being provided that highly irradiated fuel assemblies can be accommodated after a three-month decay period. Because of the heavy shielding, no limitations will need to be imposed on U^{232} content or decay time after removal of U^{232} decay daughters. The four operating cells of the facility are each 20 ft wide and have a combined length of 106 ft. The total estimated cost of the facility with minimal sol-gel vibratory-compaction equipment is $7,275,000. Facility planning is in the preliminary design stage (title 1), with Giffels and Rossetti, Inc., serving as architect-engineer. Design has been in progress since January 3, 1963, and is about 90% complete. Title 1 design for the process equipment is being prepared by ORNL.

Preparation and Properties of U(IV) Sols and of UO_2. — Nitrate- and oxalate-stabilized U(IV) sols were prepared. High-density UO_2 was prepared by precipitation with ammonia from uranous nitrate and uranous formate solutions. High-surface-area UO_2 (13.5 to 58 m^2/g) was prepared from uranous oxalate and by reduction of uranyl carbonate solutions, but the dried oxide failed to exhibit good colloid-forming properties when various electrolytes in aqueous solution were used in attempts to peptize it.

The System UO_2-ThO_2-O_2. — Equilibrium phase relationships in the system UO_2-ThO_2-O_2 are being studied because of their importance in the preparation, performance, and processing of fuels based on thorium and uranium oxides. Experiments so far have been limited to equilibrations at a constant partial pressure of oxygen of 0.2 atm (1 atm of air) and a temperature range from 600 to 1600°C. They have included all U/Th ratios from pure ThO_2 to pure UO_{2+x}.

Thorium Blanket Fluids for Future Reactors. — In studies on blankets for thorium breeder reactors, the system NaF-KF-ThF_4 appears to be the simplest system suitable for use as the blanket of a fast molten-salt breeder reactor. Results obtained so far indicate that since substitutional solid solutions are readily formed between NaF-ThF_4 and KF-ThF_4, the probability of segregation among the crystallizing phases of NaF-KF-ThF_4 is diminished.

Fission Gas Release from UO_2 and ThO_2–2% UO_2. – The effect of creep loading and temperature on release of fission gas from ceramic fuels is being studied to determine the mechanism of release under stress, especially in operating reactors. There is an enhanced release which is more pronounced the higher the temperature.

Sol-Gel Preparation and Properties of ThC_2 and UC_2-ThC_2. – Kinetic studies of the reaction of thoria and mixed thoria-urania sols with high-specific-surface-area carbon to form the respective carbides were carried out. Oxides prepared by the sol-gel process were much more reactive than oxides prepared by other means – by thorium oxalate decomposition, for example. This results in a significant lowering of the temperature at which rapid reaction occurs.

Particle Sizing of Sol-Gel Thoria and Thoria-Urania. – Thoria and thoria-urania may be sized for vibratory compaction to about 90% of theoretical crystal density by simple fracturing and ball-milling operations at the gel stage. Recycle of material is accomplished by redispersion in water.

Formation of Sol-Gel Microspheres. – Thoria microspheres of uniform size were made in the 200- to 800-μ-diam range by dispersion and gelling of thoria sol in carbon tetrachloride containing isopropyl alcohol. Production of uniformly sized spheres having diameters anywhere in the range 50 to 1000 μ appears feasible. The spheres appear suitable for use in breeder fuels of the oxide dispersion type. The same technique was applied to formation of thorium-uranium dicarbide microspheres, and several batches were made. There is some problem of sol stability when carbon is present, and this is being given great attention.

Dispersion Hardening of Thorium-Zirconium Alloys. – Hot tensile tests (800°C) were made on thorium alloys containing 5 wt % Zr and 5 wt % Zr–10 vol % ThO_2 prepared by ball milling the hydrides of thorium and zirconium, hot pressing, and extruding. Initially 800°C stress-rupture testing was planned for the Th–5 wt % Zr alloy, but the specimen failed upon loading at 4000 psi.

Irradiation of Thorium Oxide Fuels. – Sol-gel-prepared thoria-urania irradiated to 14,000 Mwd/ton showed little effect of irradiation. Fission gas retention is comparable to that of arc-fused material. Effective thermal conductivities of ThO_2–2.9% UO_2 were 0.021 w cm^{-1} $(°C)^{-1}$ at 990°C and 0.0175 w cm^{-1} $(°C)^{-1}$ at 1325°C. These values are normal.

Thoria Preparation. – Thorium nitrate and uranyl nitrate (U^{233}) were purified from a common feed by solvent extraction and made into mixed oxide by the sol-gel process. About 130 kg of mixed oxide was prepared without any reject material, although some blending of batches was necessary.

Rod Fabrication. – Eighty-nine rods were completed for the Brookhaven National Laboratory criticality experiments. Seventeen rods were rejected because of density variations of greater than ±2% of the average density along the length of the rod. Several maintenance and repair operations were necessary in the fabrication and testing equipment.

Pellet Fabrication. – Pellets for about 50 fuel rods will be needed for use in flux distribution measurements in the zero-power criticality tests to be carried out at Brookhaven National Laboratory with about 1100 sol-gel thorium-uranium oxide fuel rods prepared by vibratory compaction in the Kilorod Facility. The 50 rods will be nearly identical with the 1100 rods, but will contain right cylindrical pellets of thoria-urania rather than the compacted oxide. The use of pellets will permit the placing of flux-monitoring foils at various locations in the rods.

MOLTEN-SALT REACTOR PROGRAM

Molten-Salt Reactor Experiment. – Progress was made in the installation of auxiliary piping, instrumentation, and electrical services in all areas of the MSRE. The fuel drain tank and steam dome assemblies were fitted to the jig and prepared for installation. The coolant drain tank was installed, and installation of coolant piping was started. The reactor-cell wall penetrations for the 5-in. coolant piping were installed. The heat exchanger was fitted to the assembly jig, and the welding of fuel system piping to the heat exchanger and freeze flanges was started.

The H. K. Ferguson Company continued to install electrical and auxiliary systems.

The overall project was 78.4% complete on August 1, 1963.

Reactor Analysis. – There will be an inherent source of neutrons in the MSRE fuel salt. Before power operation, most of the neutrons will be produced by interaction of alpha particles from the U^{234} with the fluorine and beryllium in the salt. Calculations showed that with the critical concentration of uranium in the fuel, the α,n source in the 25 ft^3 of fuel salt in the core produces about 4×10^5 neutrons/sec. Spontaneous fission contributes 20 to 700 neutrons/sec, depending on the amount of U^{238}. The photoneutron source, produced from the beryllium by fission-product gamma rays, will be much stronger than the other sources after high-power operation. For example, one month after shutdown from a week of operation at 10 Mw, the photoneutron source in the core produces 3×10^8 neutrons/sec.

Analysis of accidents involving ramp additions of reactivity with the reactor initially subcritical indicates that the safety requirements for a source are satisfied by the inherent α,n source. An extraneous source will probably be used for convenience in monitoring the reactivity of the subcritical reactor during preliminary experiments and routine startups.

Calculations indicate that there is no danger of criticality in the fuel storage tank with fuel salt of normal uranium concentration, even if the salt is cooled to 20°C and the tank is submerged in water, provided that the uranium remains evenly dispersed in the salt. MODRIC calculations for the storage tank at 20°C, with an infinite H_2O reflector, showed that if the uranium were concentrated in a uniform region at the center, as by selective freezing, criticality would be reached at a concentration factor of about 4. In the drain tanks, because of the poisoning effect of the cooling thimbles, higher concentrations are required for criticality.

Development of Components and Systems. — After 2018 hr of continuous operation, the engineering test loop was shut down for examination of the graphite access joint. Samples removed from a deposit at the liquid-gas interface of the joint showed evidence of phase segregation, with collection of small amounts of corrosion products and ZrO_2. It was concluded that the phase segregation was caused by the flow and temperature conditions in the joint, and that the deposit could have been dissolved by raising the temperature and liquid level just before the loop was shut down.

The chromium content of the loop salt samples had leveled just before the loop was shut down, and there was no further increase after the loop was refilled and operated for a month.

The sampler-enricher mockup, which is attached to the engineering test loop, was operated without difficulty to isolate a salt sample under conditions expected during reactor operation. Further testing was started to improve the design of the capsule and to prove the reliability of each component. Operational procedures are being written.

The prototype control-rod-drive unit, designed and fabricated by the Vard Corporation, Pasadena, California, was received, and testing of the entire control rod assembly was started. To date, only minor changes in design have been indicated.

Testing of a simplified freeze valve for use in 1-1/2-in.-diam pipe was started. The valve differs from those previously tested in that the cooling air is channeled around the valve in a 1/2-in. by 2-in. duct, and in that the valve heater is a simple furnace characterized by high heat capacity. The valve required 50 to 100% more time for the normal freeze-and-thaw operation than valves previously tested. However, when operated under conditions simulating a power failure, the valve thawed in its normal time of 7 min, compared with 25 min for previous valves under similar conditions. The valve is simple to operate and, in general, can be made more reliable than previous valves. Testing is continuing.

Evaluation of equipment for the closed-circuit TV system, which will be used for viewing during maintenance operations requiring large openings in the reactor-cell shielding, continued. An ORNL representative observed and tried a multiple-camera monitor system used in an orthogonal array in a SNAP reactor dismantling facility by Atomics International, in Los Angeles. This system was adapted to the MSRE maintenance mockup and used to view the operations of replacing a pump. Recommendations were then made in favor of the orthogonal system rather than the stereo system because of better reliability, less operator fatigue, and a simpler installation.

MSRE Pump Program. — The modifications to the test facility for the MSRE prototype fuel pump were completed, and tests of the supports for the MSRE fuel pump were started. During heatup and at 1200°F, the supports provided the expected horizontal displacement, but not the vertical displacement. Inspection revealed signs of rubbing between various components of the supports. They were removed for modification and further testing and were replaced with the structures used previously. Additional tests were initiated to measure the concentration of undissolved gas circulating with the salt in the loop piping at ranges of temperature, pump speed, and pump tank pressure of interest to the MSRE.

A mockup was made of the new fuel-pump tank, which provides 6.3 ft^3 for thermal expansion of fuel. Water tests will be performed with it to establish the internal design, which controls the behavior of the gas and liquid in the pump tank. Stripping tests in which carbon dioxide and water, respectively, simulate Xe^{135} and salt will be made, and the concentration of undissolved gas in the circulating water will be measured.

Manufacture of one of the drive motors for the MSRE pumps is nearing completion, and its vessel is being inspected prior to final machining. The coolant pump tank is completely fabricated and is being inspected. The fuel-pump tank is nearing completion.

The Inconel centrifugal pump (PKP type) was subjected to a cold shakedown test and was installed in the hot-test facility for operation with molten salt.

Instrumentation Development. — Installation of a ball-float level transmitter on the MSRE pump test loop was completed, and developmental testing was begun during July. Initial performance was satisfactory. Testing will be continued over an extended period in order to obtain performance data under dynamic conditions approximating those to be found in the reactor system.

Thermocouple development efforts are presently being directed towards adapting laboratory techniques and procedures for fabrication of large quantities of thermocouple junctions, attachments, disconnects, and lead-wire seals to field conditions at the MSRE site. Major difficulties have been resolved, and field installation is under way.

NUCLEAR SAFETY

Fission Product Transport and Retention. — Preliminary experiments performed to determine the radioiodine-retention capacity of platinized alumina and that of potassium iodide in high-temperature air streams gave promising results. The need for materials having this property arises from the danger that, in a reactor accident involving a containment system permitting discharge of gases through a gas-cleaning system, hot oxygen-containing gases might destroy charcoal beds, which are presently considered the best material for iodine trapping.

Tests in which a bed of platinized alumina (a commercial hydroforming catalyst) containing I^{131} was exposed to air streams at various temperatures showed that this material could serve to hold iodine for several hours at temperatures as high as 525°C. At 625°C nearly 100% elution of iodine occurred in 1 hr. This material could also serve as a heat-sink to cool hot gases.

In the case of the potassium iodide bed, the I^{131} apparently exchanged with the nonradioactive I^{127} in the bed at 500°C; only a small fraction of the I^{131} was removed from the bed by exposure to flowing 600°C air for 4 hr, although slight decomposition of the bed occurred under these conditions. Further testing will be required to evaluate the relative merits of the two methods of holding iodine in high-temperature air.

Melting Experiments in TREAT Facility. — Two experiments were performed in the TREAT Facility to study the release of fission products during transient melting of UO_2. A preliminary low-energy transient of 60 Mw-sec was run on the first assembly to determine the transient heating rate. After this test, the fuel (30 g of 10% enriched UO_2) was preheated electrically to 800°C to minimize thermal shock, and a 330 Mw-sec transient was performed with a 108-msec period. In the second experiment, the fuel was preheated to 800°C and a 338 Mw-sec transient was performed at a period of 87 msec.

The preliminary transient on the first experiment showed a fission energy input of 0.75 cal per g of UO_2 per Mw-sec of reactor energy. This indicated that the fuel specimen in both experiments should have been nearly completely melted. Both experimental assemblies have been shipped to ORNL for disassembly and radiochemical analysis.

Unirradiated fuel was used in a purified argon atmosphere in the first two experiments. Future experiments will be designed to investigate the effects of transient energy and period, preheat temperature, argon-steam atmosphere, and burnup.

Release of Fission Products on Out-of-Pile Melting of Reactor Fuels. — The examination of the effect of burnup on fission-product release from aluminum-uranium alloy is continuing and will include studies of a series of MTR fuel specimens with burnup levels of 3, 9, 15, and 20%, in addition to the completed work at trace level and 23.6% burnup. Preliminary results obtained with 9%-burnup samples support the belief that most of the enhancement in release above trace level occurs after a relatively low amount of burnup.

An opportunity for a more realistic evaluation of the release from melted aluminum alloy fuel was encountered upon obtaining a fuel assembly (6.7% burnup) that had been damaged in the ORR accident of July 5, when coolant water flow through the assembly was greatly reduced by a neoprene gasket that dropped on top of the reactor core. Upon dismantling the assembly, a melted area of about 18 in.^{2} was found in the upper half of the third plate from one side. Loss of material apparently occurred only from this one plate and was determined by difference in weight to be about 13.5 g, slightly less than 10% of the total weight of the plate. Overheating of portions of all the plates was evidenced by discoloration, possibly from fuel penetration to the surface, and by a thin layer of aluminum oxide, formed by reaction of the cladding material with steam. Globules of alloy removed from the edge of the hole in the plate were photographed, weighed, and dissolved for radiochemical analysis to determine their fission-product content. This will be compared with the fission-product content of a sample of the adjacent unmelted fuel.

Samples of irradiated U-Al alloy were used as a realistic source of radioiodine in tests of the filter and charcoal arrangement proposed for use in the containment system of the ORR. The results suggested that more than 99% of the iodine released from the fuel samples in air was in the form of molecular iodine, and showed that the efficiency of two 3/4-in.-deep beds of coconut charcoal in series was greater than 99.99%.

Release of Fission Products on In-Pile Melting of Reactor Fuels. — The eleventh experiment to study the release of fission products on

in-pile melting of UO_2 fuel was performed. The flow of helium sweep gas, which was increased from 400 cm^3/min used in previous experiments to 800 cm^3/min in experiment 10, was further increased to 4000 cm^3/min in experiment 11. Other conditions remained the same as in previous experiments (3 to 9). Radiochemical analysis is complete for experiment 10 and is approximately 50% complete for experiment 11.

The percentage release values for experiment 10 were as follows:

Origin of Release	Sr^{89}	Zr^{95}	I^{131}	Te^{132}	Cs^{137}	Ba^{140}	Ce^{144}	UO_2
Fuel[a]	68.8	57.7	97.3	95.9	94.9	72.5	59.3	55.8
High-temperature zone of furnace[b]	2.1	2.8	89.5	71.5	79.7	2.8	1.0	0.29

[a]Includes miniature fuel element and holder.

[b]Includes fuel and thermal insulation; minimum temperature, 1000°C.

The results of experiment 10 indicate no marked change with increased flow rate; in general, previous conclusions regarding release have been substantiated.

Characterization and Control of Accident-Released Fission Products.— It had been observed in earlier studies that when iodine, presumed to be in the elemental form, was introduced into a filtered and purified air stream to give a concentration of about 1 mg/m^3, at least two distinct compounds of iodine were present in the air-iodine mixture. Further investigation using the diffusional deposition technique and mass spectrometry indicated that one of the compounds had a diffusion coefficient of 0.05 cm^2/sec in air and, also, that the iodine source remaining after the experiment contained compounds having masses of 284 and 338 (amu), both masses being compatible, approximately, with the diffusion coefficient of 0.05 cm^2/sec. From the diffusional deposition and allied data based on radioactivity, as much as 6% of the iodine was observed to be in the form of compounds.

In another approach to determine the origin and identity of the iodine compounds and thus their significance with regard to nuclear safety, attempts were made to prepare pure iodine using a method similar to that described by British workers, which involves the evolution of elemental iodine from melted potassium dichromate containing NaI^{131} and KI^{127}. The purity was determined by observing the behavior of the iodine in diffusion tubes. With helium as a carrier gas, a purity of 99.6% was achieved; but with air, the purity was only 98.8% and the principal impurity had a diffusion coefficient of 0.05 cm^2/sec, as had been obtained earlier. This indicated that the air was at least partially responsible for the appearance of compounds; additional work on this problem will be planned to investigate the source of the air impurity.

WASTE DISPOSAL

Clinch River Study. – Tests on the effect of the summer drawdown flows from Melton Hill Dam on the release of radioactive material from White Oak Creek into the Clinch River have been completed. Through the cooperation of TVA, water releases through the gates have simulated turbine releases expected during the summer months. Because of the peaking nature of Melton Hill Dam, water releases (in cfs) have been: 8000 from 7 to 8 AM, 16,000 from 8 AM to 12 noon, 8000 from 12 noon to 1 PM, and 8000 from 5 to 9 PM. During the higher water elevations in the Clinch River due to Melton Hill releases, radioactive material cannot leave White Oak Creek. Dyes were used to simulate the movement of the radioactive material. Dye was injected as an instantaneous line source in the Clinch River and as a 24-hr and 168-hr source at White Oak Dam. Downstream concentrations of the dye were measured at Clinch River Miles 17.5, 16.2, 14.4, 12.5, 9.1, 7.5, and 5.5 by the ultraviolet fluorescence of the dye. The times of arrival and relative magnitudes of the peak concentrations were as predicted by calculations. It was not possible to predict actual magnitude and duration of the peaks.

Mineral Exchange Studies. – Cesium sorption curves were determined for three illites in their natural state, in the potassium-saturated form, and after being heated to 600°C and cooled. The test solution was 0.1 N NaCl containing cesium ranging from 1 to 2000 mg/liter. The general shape of the cesium sorption curves for all three samples resembled those obtained from biotite and muscovite, but the curves differed significantly from each other.

The Fithian, Illinois, illite sorbed the highest amount of cesium after being heat treated. This was similar to sorption by Wyoming bentonite and a Virginia meta-bentonite which had been heated to 600°C; this illite sample apparently contained bentonite. Sorption curves for the Beavers Bend, Oklahoma, illite were similar to those for a hydrobiotite. A secondary maximum in sorption was observed for the natural material; on potassium saturation, however, this maximum disappeared. The heat-treated sample showed the lowest sorption of cesium. These results suggest that vermiculite is an impurity in the Beavers Bend sample. The Marble Head, Wisconsin, illite showed very little difference in cesium sorption after being heated or potassium saturated.

The ion exchange capacity of the Wisconsin illite was 21 meq/100 g, compared with 15 meq/100 g for the Illinois illite and 12 meq/100 g for the Oklahoma illite in their natural states. The 10-A c-axis spacing was well defined and intense for the Wisconsin and Oklahoma illites. The Illinois sample showed a very weak and diffuse 10-A c-axis spacing. Evidence of interstratified chlorite-vermiculite was present in the Oklahoma sample; feldspars may be present in the Wisconsin sample.

Disposal in Deep Wells. – Construction of the foundations for the wellhead, injection pump, and mixer cells began. The cement-blending tanks are on site, and the bulk storage tanks are in transit. Drafts of the plant operating procedures and hazards reports were prepared.

The 2000- and 50,000-gal water injection and flow-back tests have been completed, and the same fracture was reinjected with 23,000 gal, the volume of water recovered. A comparison of the initial pressure drop and flow-back rates from these three injections strongly suggests that the reinjection with the volume of water that had back flowed did not re-establish the conditions produced by the 50,000-gal injection. A substantial proportion of this water has apparently either migrated out into the wall rock, despite its apparent low porosity and permeability, or moved updip by slowly extending the initial fracture. A second series of water-injection tests using fluid-loss additives, with or without a light-weight propping agent (probably ground walnut shells), may help answer this question. The possible direction of movement of fluid out of the fracture must be considered in evaluating the fluid loss, viscosity, and setting times of any waste mixes intended for routine waste disposal.

WASTE DISPOSAL DEVELOPMENT

Foam Separation Studies. — The scavenging-precipitation foam-separation process for decontaminating low-radioactivity-level process waste water includes the following steps: precipitation of hardness by making the water 0.005 M each in NaOH and Na_2CO_3 and 2 to 10 ppm in Fe^{3+}; removal of the resulting solids in a gently stirred sludge column; and foam separation in a countercurrent column with sodium dodecylbenzene sulfonate as the surfactant. The surfactant-rich foam condensate, which also contains radiochemical impurities and solids, is recycled to the sludge column. By this arrangement there is only one waste stream, namely the sludge, which is drawn off continuously. In addition, surfactant consumption is minimized.

The performance of the sludge column is highly satisfactory. However, foam column operation has not yet been as good as was predicted from tests on the performance of a foam column under total recycle conditions.

During the first tests, at a water flow rate of 20 gal ft^{-2} hr^{-1} in the sludge column and 60 gal ft^{-2} hr^{-1} in the foam column, ORNL tap water spiked with Sr^{85} tracer was decontaminated by factors up to 8500. The average concentration of dodecylbenzene sulfonate in the decontaminated water was 5.6 ppm. Strontium decontamination factors were as high as 380 across the sludge column and 140 across the foam column. Decontamination across the sludge column was higher by a factor of about 25 than that previously observed for strontium decontamination from ORNL process waste water. Recycling of the foam condensate to the sludge column, which made the water in the sludge column about 17 ppm in dodecylbenzene sulfonate, did not seriously disrupt the mechanical operation of the sludge column. The only apparent change caused by this recycle was the accumulation of a small quantity of solids on top of the otherwise clear water above the sludge upon prolonged operation (3 or 4 days).

Decontamination of water with respect to cesium was also tested in this apparatus. The test consisted in continuous addition of −230 mesh Grundite clay (which strongly sorbs cesium) to Cs^{137}-spiked tap water at a rate of 0.4 to 0.6 lb per 1000 gal of water (in a tank, 5.5-min retention time) before the caustic-carbonate and Fe^{3+} water-softening chemicals were added. The factor for cesium decontamination across the sludge column was 5.3 to 5.9. The foam separation column did not remove any soluble cesium; however, it increased the overall factor for decontamination of cesium to 7.8 to 8.5 by removing entrained solids.

Foam Separation Engineering Studies. — Experiment shows that foams are easily condensed while passing through an orifice with upstream-downstream pressure ratios of 2 or greater. The orifices, 0.015, 0.100, and 0.250 cm in diameter, all gave residual foam volumes of about 0.1% of the inlet volumes for foams with mean bubble diameters of 0.05 to 0.08 cm. The 24-in.-diam foam column (the proposed diameter for the low-level-waste pilot plant column) was operated at flows up to 80 liters of foam per minute and 13 liters of liquid per minute. The amount of channeling appeared acceptable at 5 liters or less of liquid per minute and 50 liters or more of gas per minute; an improved liquid feed distributor will be necessary for higher liquid flow rates.

High-Level-Waste Disposal. — A neutral ceramic developed for fixation of Purex waste containing sulfate appears to meet the requirements for stability, thermal conductivity, solubility, and low melting point. Simulated Hanford FTW-65 waste solution was fixed in a microcrystalline solid by the addition of lithium, aluminum, calcium, and phosphate to the waste solution. Volume reduction from the unconcentrated waste was about 31. Sulfate loss was about 0.4%, due to entrainment. Thermal conductivity was in the range 1.3 to 1.6 Btu hr^{-1} ft^{-1} $(°F)^{-1}$ between 149 and 538°C. Leaching tests on specimens spiked with Cs^{137} gave rates which decreased to an apparently steady-state value of about 1.1×10^{-4} g cm^{-2} day^{-1} after about 30 days. The solid was mechanically strong and cracked into a few large pieces when broken, rather than scattering. The melting temperature was about 700°C. Corrosion of stainless steel in the melt at 950°C appeared to be no more severe than in air at the same temperature.

Nonsulfate Purex waste was incorporated into phosphate melts which were fluid at temperatures as low as 500°C and appeared to be true glasses. Corrosion of type 304L stainless steel at 950°C for more than 24 hr in one such melt was significant but was not highly localized.

Corrosion tests on type 304L stainless steel, Hastelloy F, and titanium 45A all showed negligible (≤0.1 mil/month) corrosion rates for 840 hr of exposure in all phases of refluxing FTW-65 waste. There was no indication of local attack on either specimen of a titanium-304L stainless steel couple.

Disposal in Natural Salt Formations. — Design and preparation of the demonstration disposal of high-level solid radioactive waste using canned ETR fuel assemblies in the Lyons, Kansas, salt mine is proceeding. The contract for the design and fabrication of the underground waste

transporter has been let. The surface facilities are being renovated, and surface grading is complete. More than 20 creep gages have been installed underground in the area around the waste shaft and the main haulageway to the site. These gages will show the effect on the stability of the area caused by increasing the haulageway height to permit passage of the waste transporter.

The Bureau of Mines has installed a hydraulic flat-jack block, about 5 ft square, for calibration of stressmeters, in the Hutchinson mine. Calibration tests of various types of stressmeters for possible used in the demonstration were nearly complete when one of the jacks ruptured. Results of the tests have not been received.

Engineering, Economic, and Safety Evaluation. — Costs of permanent storage of calcined radioactive wastes in concrete vaults and in rooms mined out of granite formations were estimated. In comparison with previously estimated costs of storage in salt mines, costs of storage in concrete vaults were five to seven times as much, and costs of storage in granite were about twice as much. This economic advantage, as well as the greater safety it is believed to offer, makes salt the preferred choice.

Vaults for storage of calcined solid wastes would be similar in their gross features to many of the concrete secondary containments used for tanks built for storage of liquid radioactive wastes, in that they would be underground structures of reinforced concrete with about 10 ft of earth cover and a floor-to-ceiling height of about 15 ft. For maximum safety, they would be sealed completely from the surface; space requirements were calculated by assuming dissipation of the heat of radioactive decay by conduction through the earth cover. Two types of concrete were considered: ordinary concrete, capable of withstanding 400 to 500°F, and "high-temperature concrete," capable of withstanding 1000°F.

Space requirements for the storage of calcined wastes in rooms in granite formations are about the same as those for storage in salt formations. However, excavating costs are higher for granite because heavier equipment is required, drilling is more difficult and slower, and costs of explosives are higher.

PHYSICAL RESEARCH

PHYSICS AND MATHEMATICS

Medium-Energy Nuclear Physics: Heavy Particles. — In continuation of work on the $C^{12}(O^{16},\alpha)Mg^{24}$ reaction, angular distributions were measured with the eight-counter array for O^{16} energies from 41.6 to 42.0 Mev. Eleven sharp minima were found, indicating a strong contribution from $\ell = 11$ partial waves. Angular distributions were obtained also at 49 Mev. Four of the 30-Mev distributions measured previously were extended to 0°. Zero-degree excitation functions for transitions to the first six excited states of Mg^{24} were obtained over a range of several Mev, starting at 29.7 Mev.

A study of the angular distribution of Li^7 from the $B^{11}(O^{16},Ne^{20})Li^7$ reaction was initiated at the Tandem Van de Graaff. Preliminary results indicate that groups corresponding to the ground state and the first and second excited states of Ne^{20} can be resolved. Typical differential cross sections at 36 Mev incident energy and 70° (c.m.) are approximately 5 microbarns/steradian.

Excitation functions for the production of F^{18} and O^{15} from N^{14} bombardment of Li^6 were measured with the 63-Inch Cyclotron. The cross sections at 27 Mev are about 100 millibarns for the F^{18} and 30 millibarns for the O^{15}.

Medium-Energy Nuclear Physics: The 86-Inch Cyclotron. — Preliminary calibration of the magnetic analysis system was completed. The location of the focal plane was determined by using a narrow slit and a single NaI crystal detector. The resolution of the system for the full-energy protons (22 Mev) was measured to be about 0.28%. This is nearly a factor of 2 better than anticipated from the design criteria of the system.

To make full use of the resolution of the magnet system, a nuclear emulsion camera was fabricated and a program of exposing and reading nuclear plates was initiated; preliminary results indicate a system resolution of 0.25%. This is an eightfold improvement over the previous system with an NaI scintillation crystal on the 24-in. scattering chamber, and a factor of 5 better than the resolution obtained with a lithium-drifted counter.

The availability of increased resolution is of particular interest in the (p,d) pickup reactions. One of the first reactions being studied with the new system is $Nb^{93}(p,d)Nb^{92}$. The final nucleus, Nb^{92}, is an odd-odd nucleus with the odd neutron and odd proton being sole occupants of their respective shell-model levels. The low-lying levels are expected to arise from the possible couplings of the neutron and proton. In our former (p,d) work the ground-state peak was found to be very broad, but no individual levels could be resolved. With the new magnet system it is now possible to resolve individual levels. This is the first time that these levels have been observed. The determination of such levels has a direct bearing on the understanding of the proton-neutron force in nuclei.

Cyclotron Operations: The Isochronous Cyclotron. — Beam deflection efficiency was markedly improved after installation of the spectrometer-type accelerating slit for defining the beam during 120° of the first revolution and after optimization of the harmonic coil currents; 50–70% of the circulating beam now passes through the electrostatic deflector channel to the internal probe. The coaxial magnetic channel was then installed, and conditions for optimizing the beam through it are being studied; 30% of the circulating beam is now obtained through the channel.

To verify machine performance, the energy of a deflected deuteron beam was measured by activation of a copper foil stack; the measured value of 35 Mev is in good agreement with calculations from magnetic field data.

Measurements of activation resulting from deuterium, molecular hydrogen, and alpha-particle beams on copper targets show that, from the standpoint of residual activity and neutron production, the molecular hydrogen beam is the most desirable one to use for machine performance tests. Dissociation of a fraction of the hydrogen beam does, however, result in high-energy particles emerging tangentially from the magnetic field; graphite shielding must be provided around the periphery of the machine.

A dee voltage regulator designed to improve beam quality by reducing the modulation of the rf voltage from 10% to about 1 part in 1000 was installed and is undergoing operational tests.

Physics of Fission. — Fission-fragment energy correlation experiments were completed for the thermal-neutron-induced fission of Pu^{239} and Pu^{241}. Two silicon surface-barrier detectors were used to detect the fragments; the correlated pulse amplitudes were sorted and were summed into a 128 × 128 channel matrix. Appropriate transformations yielded the fragment mass distributions. It was found that near symmetry, the difference of 2 amu between Pu^{241} and Pu^{239} appears predominantly in the light fragment; for very asymmetric fission, this difference appears approximately equally for the light and the heavy fragment.

High-Voltage Experimental Program: Neutrons from the Proton Bombardment of Li^6, Li^7, Be^9, B^{11}, and O^{18}. — The total neutron yield from proton bombardment was measured for Li^6 from threshold to 14 Mev; for Li^7, Be^9, and B^{11} from 4 to 14 Mev; and for O^{18} from 3.5 to 10 Mev. The $Li^6(p,n)Be^6$ reaction Q measured in this experiment is -5.05 ± 0.05 Mev, giving a Be^6 mass excess of 18.36 ± 0.05 Mev. The neutron yield from Li^7 indicates more neutrons at higher energy than can be explained on the basis of the (p,n) reaction alone; these are ascribed to multiparticle breakup. An anomaly in the Be^9 + p neutron yield curve is ascribed to a threshold for production of neutrons by a state in B^9 at 4.04 Mev. The $B^{11}(p,n)C^{12}$ reaction results confirm previous level structure in C^{12} and a new level at E_p = 11.85 Mev (energy of excitation in C^{12} of 26.81 Mev). The O^{18} + p neutron yield shows 21 "resonances" (energy of excitation 11.4 to 16.8 Mev).

High-Voltage Experimental Program: $B^{11}(p,\alpha)2He^4$ Reaction. — A preliminary study of the reaction $B^{11}(p,\alpha)2He^4$ was made for proton energies on and below the well-known 163-kev resonance by use of the Cockcroft-Walton accelerator. Enriched carbon-backed targets were used which were

approximately 20 kev thick to the proton beam. Alpha spectra were measured for laboratory angles of 35, 50, 90, and 135° at bombarding energies of 147 and 180 kev. In addition, the spectra of alpha particles at 90° in coincidence with alphas at 35, 50, 90, and 135° were measured at bombarding energies of 147 and 180 kev. With the exception of the 90-50° and 90-90° correlations, all these spectra differed markedly at the two different energies, that is, on and below the 163-kev resonance. A preliminary analysis indicates that the breakup mechanism below the resonance is a sequential two-body process which proceeds via Be^8, while the breakup process on the resonance is a nonsequential three-body one.

Stable Isotopes Development. — The two inhomogeneous-field 255° separators installed in the XBX vacuum tanks are being used to process cadmium. The cadmium isotope enrichments are slightly higher in these 20-in.-radius separators than in the 48-in.-radius separators formerly used. Average ion currents are 25 ma, and assays show that Cd^{111} has reached 97.1% isotopic purity.

Sector magnetic fields of the inhomogeneous type are being studied for use in the isotope separations program. An attempt is being made to determine, at least qualitatively, the effect of fringing fields upon ion trajectories through the system. Estimates of the values of the effective field components are made by combining the field gradients from the fringing field in front of a uniform-field magnet with those of an inhomogeneous magnetic field with no fringing, assuming that the field lines lie in planes parallel to the z axis as they fringe away from the magnet poles. Ion orbits based on these approximations are being computed and plotted with the CDC-1604 computer.

Miniaturization of existing calutron ion sources would provide many advantages in second-pass separations and anticipated radioisotope separations where about 1 to 2 g of feed material is available. A one-half-scale model of the conventional M-16 calutron source has been constructed and is now available for testing.

An ARL-Dietert reflecting grating spectrograph is being used to establish, to a limited degree, the atomic and ionic components within the calutron system. Observations made at both the source and receiver positions show an abundance of neutrals and low-energy ions in each location. In the source area, some small percentage of the ion energies calculated from the observed Doppler shifts is in excess of the applied accelerating potentials. These ion energies, if real, could be of prime importance in introducing contamination into isotopic samples.

Special Separations. — The estimated weights (based on monitored ion currents) of separated plutonium isotopes and the ranges of enrichment achieved are as follows:

Isotope	Estimated Weight (g)		Purity Range (%)
	For Period	Cumulative	
Pu^{239}	12.9744	62.1781	94.79–95.27
Pu^{240}	23.1686	109.1582	96.63–99.30
Pu^{241}	6.1931	27.9822	88.61–95.88
Pu^{242}	2.9123	13.8605	84.22–91.19
Total	45.2484	213.1790	

A study has been made of a cocurrent liquid-liquid extraction process in which tributyl phosphate is used for recovery of uranium from calutron wash solutions. Spectrographic analysis of the recovered uranium showed satisfactory decontamination from iron, copper, and nickel, which are the main impurities.

Three returned batches of enriched uranium isotopes (U^{233}, U^{234}, and U^{235}) were recovered, purified, and made available for research purposes. Quantities of neptunium fluoride, americium fluoride, and plutonium fluoride ranging from 100 to 200 mg were prepared by a wet chemical method. Two plastic scintillators were each plated with ~1.0 μc of U^{232}, and the interior surfaces of four small nickel tubes were plated with Np^{237} to a thickness of ~0.01 mg/cm^2.

CHEMISTRY

Instrumental Methods for Continuous Chemical Analysis. — The seventh in a series of models of high-sensitivity recording flame spectrophotometers has been designed and put into service. A Beckman oxygen-hydrogen burner serves as the source unit for emission flame photometry, with provision for the addition of a source unit with a long optical path for atomic absorption flame photometry. A Jarrell-Ash Ebert scanning spectrometer with a linear reciprocal dispersion of 16 A/mm provides a resolution of at least 0.2 A (10-μ slits) in the first order and eight scan rates from 2 to 500 A/min over the range from 2700 to 8700 A. For spectrally uncomplicated samples, wide slits can be used that increase the photoelectric sensitivity; even with 500-μ slits, the spectral band pass is only 8 A. At the entrance slit, the light beam is chopped at 320 cps by a heavy perforated disk driven by a synchronous motor. The wavelength range from 2000 to 8700 A can be covered with one multiplier phototube, a type EMI 9558 AQ; or one of several phototubes can be used, each of which covers a more limited range with somewhat greater sensitivity, such as types EMI 6256 B and ITT FW-118. Reliable, high-quality commercial power supplies and operational amplifiers reduce the cost and simplify the construction and maintenance. The phototube is directly coupled to a current amplifier followed by two ac-coupled voltage amplifiers tuned to 320 cps. The amplified signal is rectified by an operational amplifier system. Chopping provides zero stability even with infrared-sensitive phototubes which have a variable, high, and temperature-dependent dark current. With an absorption source unit, chopping would enable measurements by the atomic absorption method even for those elements which have flame emission intensities that are not negligible with respect to the intensity of the line source. Either of two modes of flame emission measurement can be used. The emission can be recorded as a function of time by means of a chopper-stabilized voltage amplifier, which is provided with variable damping and a zero set control for flame background compensation, that follows the detector. An unmodified potentiometric chart recorder having a response time of 1 sec is provided so that relatively rapid scanning rates can be used. Alternatively, while the instantaneous emission signal is being recorded, the signal can be integrated by a chopper-stabilized integrator during a fixed time interval. At the end of the integration interval, the timer automatically disconnects the recorder from

the instantaneous signal long enough to record the accumulated integral. Monitoring during integration assures that no artifacts occurred during the integration period, so that integrating over a fixed time interval is valid. The integrator is a better means of averaging the signal when work is being done at very high sensitivity when noise due to flame fluctuation is high. Elements that have been analyzed so far by emission photometry include Mg, K, Na, Ca, Li, Sn, Sr, Ba, Cd, Rb, and Cs. The instrument has high resolution and is stable and very sensitive.

Analysis of Molten Salts by Electrochemical Methods. — In a continuing study on the voltammetry of reducible species in molten fluoride salts, emphasis was placed on anodic stripping voltammetry.

Anodic stripping voltammetry is a method whereby a metal is deposited on an indicator electrode and then removed anodically under controlled conditions. This technique was applied to iron at a concentration level of 265 ppm in a molten mixture of LiF-NaF-KF at 500°C. Preliminary tests revealed that this approach may prove valuable for analyzing molten-salt systems for trace quantities of metal impurities.

By controlling such variables as electrode area, potential of the electrode during the plating cycle, plating time, and the rate of voltage scan for the dissolution process, the area under the stripping curve is related to the concentration of the metal in the melt. The method is at least ten times as sensitive as conventional voltammetry.

Stripping curves for iron were recorded at a pyrolytic graphite electrode encased in boron nitride and also at tungsten, platinum, and silver indicator electrodes. The curves, which are peak-shaped, were generally well defined. The half-peak potential occurs at approximately −0.5 v vs a platinum quasi-reference electrode.

Evidence of alloy formation was observed when the iron was deposited on a platinum electrode. For short plating intervals, of the order of 1 min, the deposited iron was not stripped off the electrode at −0.5 v but instead at approximately +0.1 v. For longer plating intervals, however, the characteristic curve at −0.5 v did appear, and the area under the curves was observed to increase with plating time. On the other hand, the area under the curve at +0.1 v did not appreciably change with longer plating periods. It appears, therefore, that the first layers of iron form an alloy with the platinum. Following a probable surface saturation effect on the electrode, the additional iron which is then deposited does so as pure iron. From these observations, platinum would appear to be the least preferred of the electrodes tested for adapting anodic stripping techniques to the determination of iron in molten fluoride salts.

CHEMISTRY RESEARCH

Thorium Oxide Studies. — Data from an ultracentrifugation study of a 1 M thoria sol of particles having a BET (nitrogen adsorption) specific surface area of 52 m^2/g indicate that a nitrate ion occupies 31 A^2 of thoria surface. For this thoria, therefore, the nitrate equivalence is 2.8×10^{-4} mole of nitrate ion per gram of thoria. For comparison it may be noted that M. E. Wadsworth *et al.* at the University of Utah found about 15 A^2 to be occupied by silicic acid on thoria. The "true" hydrogen ion

concentration of solution in equilibrium with the above sol at the nitrate equivalence point was calculated to be 3.6×10^{-3} M. The calculated pH of the solution, when corrected for a minor activity coefficient deviation from unity, is 2.47. The pH of the sol read on a pH meter using a glass measuring electrode and calomel reference electrode was 3.9. The difference is attributed to the "suspension effect" and thoria adsorption on the glass electrode. As the ratio of moles of adsorbed nitrate ion to thoria surface area drops to about 78% of the foregoing equivalence value, the equilibrium hydrogen ion concentration drops to 5.0×10^{-4} M. The gel produced by centrifugation ceases to be dispersible to a true, stable colloid, and the acid concentration in the centrifugate rises to 6×10^{-4} M.

Low-temperature sintering properties of thoria gels are of basic importance to an understanding of how the sol-gel process produces dense ThO_2. The sintering properties of thoria gel may be related to the way water is held by the gel. Equilibrium and kinetic studies by Wadsworth et al. on gel dehydration indicate that water held by the gel in the temperature range from room temperature to about 140°C is hydrogen-bonded in a state very similar to that of liquid water. The principal difference seems to be that water in the gel is somewhat less ordered (slightly larger entropy) than it is in liquid water; further, the equilibrium temperature at 1 atm partial pressure of water vapor is 137°C instead of 100°C. In order for thoria to adsorb water by hydrogen bonding in a manner similar to that in liquid water, it is postulated that surface species such as hydroxyls, nitrates, and possibly carbonates are the adsorption sites at temperatures up to 140°C. The dehydration reaction is first-order with respect to that fraction of water remaining at temperatures up to about 140°C.

Protactinium Chemistry. — The high radiation level of the protactinium which was irradiated to prepare U^{232} and the unavailability of hot cell facilities for purifying it have delayed its processing. Meanwhile, further laboratory studies are being carried out on protactinium separation chemistry. At present 1 to 2 g of the Pa^{231} is in a semipure state as the oxide. About 46 g is present in 10 liters of 8 M HCl—0.6 M HF.

The proposed purification involves complexing the fluoride present in the solution with Al^{3+} and loading both the Pa^{231} and the trace of U^{232} as chloride complexes onto an anion exchange column. Experiments with 0.5-ml samples of the feed indicate that some dilution of the feed will be necessary to prevent precipitation of aluminum salts. The process tentatively proposed is to mix 1 volume of feed (8 M HCl—0.6 M HF) with 1.5 volumes of 6 M HCl—1 M $AlCl_3$ immediately before loading onto the anion column.

A series of extractions with di(2-ethylhexyl)phosphoric acid was carried out to determine whether any significant separations could be achieved by this procedure. The results indicated that there was no separation of protactinium from uranium, thorium, or niobium. The possibility of separations in a scrub or stripping step has not been investigated.

Some progress has been made in the adjustment of the feed solutions to allow the sorption of thorium on a cation exchange column without any sorption of protactinium. In one series of experiments, diluting the feed to 2 M HCl and adding one mole of boric acid per mole of hydrofluoric acid resulted in sorption of 63% of the thorium onto a Dowex 50 column without any sorption of the protactinium. Work is continuing on this problem.

Since protactinium and niobium can be separated by chromatographic elution from a Dowex 1 column, a feed containing approximately 3 mg of protactinium and 7×10^6 gamma counts per minute of $Zr\text{-}Nb^{95}$ was loaded onto a 3-ml column and eluted with 8 M HCl–0.2 M HF. The resulting product contained more than 90% of the protactinium and no detectable $Zr\text{-}Nb^{95}$.

Attempts to precipitate protactinium from the feed by dilution with water were unsuccessful. Samples, both with and without $AlCl_3$, were diluted to 2 M HCl. These were then centrifuged, and the supernatant was analyzed. No significant change in alpha or gamma activity was noted.

Equipment Decontamination. – Fission-product deposition samples that had been exposed in an ORR poolside experiment to helium contaminated with fission products from neutron-irradiated uranium carbide–thorium carbide fuel spheres were found to have surface deposits relatively low in barium and iodine. Also, little iodine has been found so far in deposits in the loop, although the amounts of barium were relatively high. Explanations have not yet been found. In laboratory tests, however, Ba^{140} (added as chloride solution) has so far been virtually nonvolatile from graphite in helium or hydrogen at 1100°C, and iodine has not been appreciably deposited on steel above about 400°C.

An inexpensive new method for the low-corrosive decontamination of carbon steel at room temperature is under study. The method employs a preliminary 5-min treatment of the steel with 0.02 M oxalic acid to dissolve rust and condition the surface, followed by gradual addition of hydrogen peroxide up to about 0.05 M during the next 3 to 5 min. Total corrosion is about 0.05 mil, with decontamination factors of 400 to 800 for $Ba\text{-}La^{140}$ baked on at 400°C.

Pyrolysis and Radiolysis of Organic Liquids. – A material balance, within 10%, was achieved for hydrogen gas produced in the pyrolysis of biphenyl at 425°C to 0.25% decomposition. It was assumed that the net reactions caused hydrogen production during polymerization and consumption during benzene formation. Trace amounts of methane and ethane were not considered in the calculations. No deuterobenzene was formed during pyrolysis of biphenyl in the presence of deuterium gas, implying that molecular hydrogen is not involved in the mechanism for benzene production. The rate of formation of hydrogen and benzene during pyrolysis was not significantly different in the presence of water vapor (0.25 wt %). The amount of hydrogen and benzene formed upon the pyrolysis of biphenyl at 438°C for 12 hr was approximately three times that produced at 425°C in the same time interval, indicating that the temperature coefficient for the reaction is not very different from expectations.

Properties of Aqueous Solutions: Species and Thermodynamic Properties. – The analytical centrifuge has been successfully used in the past for measurement of activity coefficients of salts in dilute solutions. However, application in the concentrated range with the interference optical system, which is more accurate than schlieren systems, has not been feasible because the large difference in effective light path between solution and solvent washed out the fringes, at least with cells of currently available thickness and light sources in common use. This difficulty has been overcome by putting a solute of known activity coefficients (e.g. HCl) in the background compartment at a concentration such that the

initial refractive index of solution and solvent is approximately the same. The background distribution, and therefore its contribution to the refractive index pattern, can be computed.

With this technique, activity coefficients of $BaCl_2$ were measured in the range 0.6 to 1.6 m. An equation for log $\gamma_{\pm BaCl_2}$ derived by a least-square fit of several runs predicted values for the osmotic coefficients of this solute which fell within the scatter of Robinson's data. In interpreting the results to this accuracy it was found necessary to correct for the effect of pressure on the refractive indices of solution and background by the Gladstone-Dale or the Lorenz-Lorentz equation, with estimation of compressibility by the methods and data of Gibson.

Radiation Chemistry. — The ceric reduction yield in the ceric dosimeter is generally considered to be the same in aerated and deaerated solutions. It has been found, however, that if the solution is purged with helium during irradiation the cerous yield $G(Ce^3)$ is lowered by about 0.2. Purging with air during irradiation does not change the yield. Two explanations for the lowering of the yield are suggested: (1) a reducing intermediate is swept out of solution at low oxygen concentration and/or (2) an intermediate which normally reduces ceric ion is converted to an oxidizing species at low oxygen concentration.

To check the first suggestion, another well-studied system was purged with helium during irradiation to see whether the yield in this system was also changed. The ferrous dosimeter was chosen, and the deaerated oxidation yield was found to be unchanged by helium-purging. From this it was concluded that sweeping an active intermediate out of solution was not the correct explanation.

The second suggestion was considered in some detail. A reaction which may occur only at very low oxygen concentration and which fulfills the proper requirements is the following:

$$H + H_2O_2 \rightarrow H_2O + OH \ .$$

If this reaction is enhanced to any extent in helium-purged solutions, the cerous yield will be lowered. The lowering to be expected by this reaction is expressed by the following equation:

$$\Delta G(Ce^3) = 4G(H) \frac{k_1[H_2O_2]}{k_1[H_2O_2] + k_2[O_2] + k_3[Ce^4]} \ .$$

This equation was evaluated using steady-state kinetics and published reaction rate constants. The magnitude of the lowering suggests that the pertinent competition with the reducing species may involve the hydrated electron rather than the hydrogen atom, even though the solution is strongly acid. Also, for lowering to occur, the rate constant between Ce^4 and the reducing species needs to be $\sim 10^4$ liters mole^{-1} sec^{-1} or less. These implications were not expected, and additional work is required before this lowering in the cerous yield can be satisfactorily explained.

High-Temperature and Structural Chemistry. — Because of the analogy in electronic structure of the hypothetical diatomic alkaline earth metal subions $(Ca_2)^{2+}$ and $(Ba_2)^{2+}$ with the diatomic alkali metal molecules Na_2

and K_2, the following prediction had been made: In the molten barium halide—barium (BaX_2-Ba) systems, the rate of increase of electrical conductivity was expected to increase with metal concentration as in the KX-K systems (relatively unstable K_2 molecules), whereas in the CaX_2-Ca systems, like the NaX-Na systems (relatively stable Na_2 molecules), it had been found to decrease. This increase has now actually been observed in both the $BaBr_2$-Ba and the BaI_2-Ba system, indicating the lower stability of the $(Ba_2)^{2+}$ ions compared with that of the $(Ca_2)^{2+}$ ions. The SrX_2-Sr systems (X = Cl, Br, I) were found to resemble the CaX_2-Ca (and NaX-Na) systems.

Physical Chemistry of Gaseous Thermodynamic Working Fluids. — Thermal transpiration experiments with helium and argon have been completed. In each case, it was necessary to employ a fictitious temperature gradient in order to reconcile theory with experiment.

A prototype two-bulb gaseous diffusion cell has been constructed. Only one experimental run has been completed thus far, on the system He-Ar, but the result is in accord with values reported previously for this system. Furthermore, the reasons for the small discrepancies which do exist are known and can easily be corrected.

Target Preparation. — Magnesium films (21 mg/cm^2) and scandium films (30 mg/cm^2) have been deposited by vacuum evaporation on copper plates for use in cyclotron studies. The backing was prepared by degreasing, cleaning with dilute hydrochloric acid, and etching by glow discharge. The evaporation of both magnesium and scandium required a vacuum of 5×10^{-6} torr. Cooling of the substrate to ~15°C was necessary in the scandium evaporation, but no special treatment of the substrate was needed during the magnesium evaporation. The magnesium itself was wiped clean between evaporations to maintain a stable, adherent film.

Since present methods of elemental evaporation using resistance heating or electron bombardment have condensed only ~1% of the initial material into useful film, studies are under way to improve this efficiency. One method of evaporation consists in resistance heating with 3 in. of 1/4-in. tantalum tubing pinched off at each end and with a 1/4-in. hole drilled in the top center of the tube for evaporation of the material. In the second method, electron bombardment is used to heat a tantalum crucible, 1/4 in. in diameter and 1 in. high. Three runs by each method have been completed. During each run, a total of 32 mg of silver was evaporated onto a 2-in.-diam disk of polished stainless steel which was located 3 in. from the crucible. The material deposited by the first method averaged 4.2 mg or 13%; material deposited by the second method averaged 9.8 mg or 30%.

A target containing a ratio of 128 μg of nitrogen to 8000 μg of titanium was fabricated by the tritium sorption method, that is, introducing nitrogen into a chamber during heating of a platinum backing which contained a layer of titanium. Investigation will be continued to produce a higher yield of nitrogen in such targets.

Transuranium-Element Processing

Chemical Process Development: Separation of Curium from Americium at High Activity Levels. — Separation of curium from americium by chromatographic elution from a spring-loaded cation exchange column with ammonium

α-hydroxyisobutyrate at activity levels of 50 w/liter was achieved. Two milligrams of Cm^{242} and 4 mg of Am^{241} were loaded on a 20-ml column of Dowex 1-X8 (−400 mesh) resin at 80°C. The curium and americium were eluted with 0.4 M ammonium α-hydroxyisobutyrate at pH 4.2 at a flow rate of 0.3 ml cm^{-2} min^{-1}. Gassing problems were controlled, and separation of the bed was prevented by spring-loading the resin column. A 90% recovery of curium contaminated with 10% of the americium was accomplished.

Separation of curium from americium was also accomplished by chromatographic elution from a spring-loaded anion exchange column with lithium nitrate at activity levels of 100 w/liter. The characteristic cherry-red glow from the activity was observed to separate into two bands — a white and a red. The white glow was associated with the curium; the red was due to americium excitation. The separation factor was 1.87, and the results indicate that a complete curium-americium separation could be achieved with about 1% cross contamination.

Dissolution of Prototype HFIR Targets. — Four unirradiated prototype HFIR targets were processed for plutonium recovery. These targets consisted of a compacted mixture of high-fired PuO_2 and aluminum powders. The aluminum was readily dissolved in 1.2 M NaOH—1.5 M $NaNO_3$. Plutonium losses during aluminum dissolution averaged 0.7 mg/liter (0.002%). The PuO_2 was dissolved in 14 M HNO_3—0.2 M HF under reflux. The plutonium product, concentrated to 24 g of plutonium per liter, contained 0.02 g of aluminum per liter of solution. This method may be useful for reclaiming Pu^{242} from HFIR targets that do not meet specifications for irradiation.

Corrosion. — The susceptibility of Zircaloy-2 to hydrogen pickup in certain boiling acid solutions was further substantiated. In previous tests, 125-mil-thick specimens of the alloy contained 89 ppm of hydrogen after 1000 hr in boiling and oxygenated 6 M HCl and 440 ppm after a similar period in boiling and oxygenated 6 M HCl—0.1 M HNO_3 solution. The heat initially contained between 38 and 53 ppm of hydrogen. In the present tests, a different heat of the alloy in the form of 10-mil-thick strip was used; the initial hydrogen content was 260 ppm. After a 1000-hr exposure, the bulk hydrogen content of a specimen in boiling and oxygenated 6 M HCl was 5300 ppm, whereas 2600 ppm was found in a specimen exposed for 1000 hr in boiling and oxygenated 6 M HCl—0.1 M HNO_3. Both specimens underwent brittle fracture during bending after test.

Zircaloy-4 containing less than 20 ppm of nickel corroded ten times as fast as a 125-mil-thick heat of Zircaloy-2 in boiling and oxygenated 6 M HCl solution. After 1000 hr, the Zircaloy-4 rate was 22.8 mils/yr, as compared with 2.4 mils/yr for Zircaloy-2. The bulk hydrogen content of the Zircaloy-4 specimen after test was 130 ppm, as compared with an initial value of 19 ppm. The hydrogen content of the companion Zircaloy-2 specimen was 89 ppm after test, whereas the initial content varied from 38 to 53 ppm.

METALLURGY AND MATERIALS

Preparation of Pure Materials. — A 100-g sample of highly purified and dehydrated LiF containing 99.99 at. % Li^7 and a 288-g single crystal

of LiF containing 79.28 at. % Li^7 were produced and delivered to J. W. Cleland, who loaned them to R. O. Pohl of Cornell University.

The Li^7F will be used at Cornell in attempts to grow a single crystal by the Kyropoulos technique. The crystal containing 79.28 at. % Li^7 will be cleaved; a portion will be retained for physical measurements at Cornell, and the remaining material will be returned to ORNL for analysis and further distribution.

Research and Development on Pure Materials. — The Research Materials Information Center has coded and filmed its collection of papers, abstracts, preprints, book chapters, and reports on methods of preparation of high-purity solid-state research materials. The collection covers the literature from 1957 to the present and includes, besides preparation methods, pertinent peripheral information such as phase equilibria, diffusion and self-diffusion, melting points, and vapor pressures. Four 100-ft reels of 16-mm coded microfilm contain a total of about 9000 pages of documents ranging from 1 to 200 pages each. Long documents are coded in sections for "pinpoint" searching.

Spectroscopy of Ionic Media. — Spectra of tetrahedral NiX_4^{2-} (X = Cl, Br, I) were measured in several organic solvents with an exceptional degree of completeness; most of the spin-forbidden d-d transitions were observed, and the charge-transfer transitions were measured to 35,000 cm^{-1} for the I complex, to 40,000 cm^{-1} for the Br complex, and to 45,000 cm^{-1} for the Cl complex. The spectra were measured for several liquid media. Compilation of these data and a detailed comparison with experiment (a substantial undertaking) are now in progress. For the comparison with theory a code was written which diagonalizes the Liehr-Ballhausen matrices to give the eigenvalues and eigenvectors. Preliminary optical evidence was obtained for tetrahedral $CuCl_4^{2-}$ in several liquid media; theoretical analysis of the spectra is under way. A spectrophotometric method has been devised to measure the rate of the reaction

$$4NO_2^- + ClO_4^- \rightarrow 4NO_3^- + Cl^-$$

in fused lithium perchlorate as a solvent. The rate is pseudo-first-order and quite sensitive to several common impurities such as water.

Fundamental Investigation of Radiation Damage in Solids. — Recently it was discovered that ultrasonic waves of finite amplitude can be used to study the anharmonicity of solids. Experiments with several metals have shown that a large-amplitude ultrasonic wave becomes distorted as it progresses through the solid. The distortion is measured by determining the amplitudes of the harmonics generated in the material as the initially sinusoidal wave progresses. It has been verified that the amplitude of the second harmonic is proportional to the square of the amplitude of the fundamental, in accordance with theoretical expectation. Other aspects of the theory are being investigated and extended.

The rate at which the harmonics are generated depends on the magnitude of the second-order (ordinary) and the higher-order elastic constants (principally third-order). Thus, measurement of the harmonics for various orientations of a crystalline solid can lead to information about the

higher-order elastic constants. The results of these studies should contribute to an understanding of radiation damage processes and to a determination of fundamental properties of solids (e.g., interatomic potential functions).

CONTROLLED THERMONUCLEAR RESEARCH

Plasma Physics. — Experiments with the Ioffe technique for stabilizing magnetic-mirror machines were made on a very small scale. Four- and six-pole Ioffe fields were applied to a plasma formed at the intersection of a stream of gas atoms and a stream of electrons. A Ioffe field of only 5% of the main field, as measured at the boundary of the plasma, produced striking results. On the average, plasma density on the axis of the apparatus increased by a factor of 4, plasma noise decreased by a factor of 4, and radial plasma loss was reduced. Stronger Ioffe magnetic fields produced no further improvements in plasma stability. These experimental observations differ from those of Ioffe, where a weak Ioffe field decreased plasma containment, and a Ioffe field roughly equal to the main field produced optimum plasma containment.

Previous time-of-flight velocity measurements of ionic sound-wave pulses crossing the gap between two electrodes in a plasma gave wave velocities in reasonable agreement with theory. More recent experiments allow the pulses to be observed in the space between the two electrodes. Thus the position of a pulse is observed as a function of time, and the sense of direction of the motion of the pulse is determined.

DCX-2 Facility. — Since the last report, DCX-2 has been out of operation for cleaning and with a succession of troubles, except for two brief runs on July 11 and 12. These runs were made with the injector retracted upward 3 in. in an attempt to increase the volume available for trapped plasma. The field changes necessary to cause the beam to circulate through the plasma region with proper pitch from this new injection point were not made correctly. The performance indicated that the beam was not being injected properly. It was decided to shut down for a magnetic scan, but before the scan could be made, the injector developed a short from helical winding to ground and had to be moved for repair. Most recent attempts to operate have been thwarted by trouble in the 600-kv injector supply.

Vacuum. — The construction of a gas-leak device capable of yielding steady leaks of less than 10^{-4} torr liter sec^{-1} was completed. This facility is part of the apparatus for the ultrahigh-vacuum sorption studies. The assembly of the 15-ft^3 vacuum chamber for this work was begun.

Two additional bakeable flange designs with metal gaskets have been tested, a hollow O-ring type and a solid copper-wire type. The hollow-O-ring test has yielded the lowest leak rate and the greatest dependability ratings of the four systems studied to date. The requirement of metal flow in order to seal this type of gasket assembly has been demonstrated for the solid-copper-wire style, for which sealing was not accomplished until it was heated to above 200°C.

Four commercial block valves (3/4-in. size) were tested for seat helium leakage and permeation. Only two of these valves yielded total permeation rates of less than 2×10^{-10} atm cc sec^{-1} of helium after 10 min

exposure to 1 atm helium pressure. The base pressure and pumping speed of a new-design mechanical vacuum pump were measured and found not to meet the claimed capabilities.

Magnetics and Superconductivity. — Magnet coils for DCX-1 are being calculated which would allow raising the center field from 10 to 12 kilogauss and working with mirror ratios between 1.5 and 3. Field studies for a redesign of the ELMO facility are under way. New computer codes were written which allow the calculation of single-particle orbits for DCX-1 and DCX-2 fields modified by Ioffe bars. Mirror coils for a new ion-source test facility have been designed.

The new Magnet Laboratory has been put into operation. Magnets with a total power of 10 Mw are presently installed. The Magnet Laboratory will serve for the development of conventional magnet coils and for experiments on superconductivity.

Experiments on flux trapping in soft superconductor tubes were made which showed that a drastic revision of the accepted theories of the energetic stability of persistent currents is necessary. Measurements of critical fields and currents verified fully an analysis made of the performance of soft superconducting solenoids. A new test facility for determining magnetic moments of hard superconductor samples is operating satisfactorily. Work on special types of micromagnetometers is progressing.

BIOLOGY AND MEDICINE

TERRESTRIAL AND FRESH-WATER ECOLOGY

Leachability of Cs^{137} in the Mull on the Floor of a Tagged Tulip Poplar Forest. — Radiation ecology studies of radiocesium movement are continuing on a tagged tulip poplar stand. The availability of cesium in the soil for plant uptake and for transport by ground water was estimated from soil samples. Leachings were made on 50-g soil aliquots from a composite of twenty 1-dm^3 samples randomly taken throughout the forest floor after the first growing season. This forest soil (Emory silt loam) is a deep, well-drained, permeable soil that is slightly acid in reaction (pH 6.0–6.5) and relatively high in organic matter. It has developed in colluvium transported from Knox dolomite. The dominant clay mineral is kaolinite. Leachability of Cs^{137} was determined by leaching four 50-g (air dry) soil samples with tap water, 1 M $NH_4C_2H_3O_2$, 0.5 N HCl, 1 N HCl, and 1 N HNO_3. The results indicate that 0.3% of the total radiocesium was in soil solution (leachable with tap water); 17% was extractable with 1 N $NH_4C_2H_3O_2$ and would normally be considered exchangeable or immediately available to plants. The effect of boiling in 1 N HNO_3 for 10 min suggests that almost half of the radiocesium was in "easily weatherable" form. The quantity extracted with $NH_4C_2H_3O_2$ compares with 11% extractable with $NH_4C_2H_3O_2$ reported for a similar kaolinitic colluvial soil (Witherspoon, 1962). The greater leachability of the above soil is attributed largely to the increased content of organic matter. Also, the time of collection of samples may influence the amount extractable by $NH_4C_2H_3O_2$.

Microbial Masses on Decomposing Leaves. — In previous reports concentration factors of radionuclides in microbial tissues on various substrates as well as the effects of environmental conditions on these factors were discussed. In other reports the numbers of microbial colonies cultivated from various natural substrates on artificial media were given. Present work is aimed at the interpretation of the colony counts in terms of microbial masses by comparing colony counts with direct microscopic measurements of microbial populations. Combined knowledge of microbial masses and concentrations of radionuclides in microbial tissue will enable prediction of accumulation of radioactive materials in the microflora.

Direct estimates of fungal masses on various substrates fluctuated less than bacterial masses. The same result was found for fungal and bacterial numbers obtained by colony counts from identical substrates. Fungal masses on oak leaves were in general less than one order of magnitude higher than bacterial masses, whereas in the corresponding colony counts fungal counts were about one order of magnitude lower than those of the bacteria. Both direct microscopic counts and colony counts of fungi on oak leaves increased during leaf decay. Direct counts as well as colony counts of both fungi and bacteria were higher on tulip poplar leaves than on oak leaves. Exclusion of the microfauna in decaying oak leaves by addition of naphthalene did not affect direct and colony counts of fungi, but did result in high direct counts of bacteria from 8 to 14 weeks after application of the insecticide. This high bacterial count was not clearly reflected in the corresponding colony counts of bacteria on naphthalene-treated leaves.

Biological Half-Life for Radiocesium in Aphids. — Experiments have been conducted to determine the biological half-life for radiocesium in insects with sucking mouthparts, since previous studies have concerned only those with chewing mouthparts. A different class of elimination rate relationships might be expected because of the physiological peculiarities of insects with sucking mouthparts. *Aphis fabae*, the bean aphid, was used in these studies. Cut twigs of nasturtiums (*Tropaeolum majus*) infested with aphids were made radioactive with Cs^{134} in a water solution. After three days of feeding, approximately 30 aphids were transferred to each of three unlabeled nasturtium twigs, and the twigs plus aphids were counted through time to estimate biological half-lives. This procedure of counting aphids and plant together was necessary since removal of aphids from plants may disrupt their normal feeding activities for hours. Estimates of biological half-lives at 8, 18, and 28°C were 42, 23, and 11 hr, respectively. Thus these elimination rates follow a Q_{10} relationship, showing a doubling of metabolic rate for each 10°C increase in temperature. During the experimental period an identical procedure was followed in a series in which young born were removed before counting. No difference in elimination rates could be demonstrated, suggesting that production of new aphids is not a major loss of cesium in these animals. These biological half-lives are longer than would be predicted for animals of this size (average live weight, 0.51 mg) from previous experiments with chewing insects. In these aphids cesium may be conserved with other minerals by the filter-chamber mechanism.

Gamma-Ray Dosimetry in Cs^{137}-Tagged Tulip Poplar. — In an attempt to ascertain the internal radiation dose delivered to plants containing radionuclides, and investigate the possible use of dosimetry as a means of

tracing radionuclide movement, metaphosphate glass-rod dosimeters were placed in components of a Cs^{137}-inoculated tulip poplar tree. This tree, inoculated with 11.5 mc during May 1962, measures 15.5 cm in diameter at breast height and is 17 m in height. Dosimeters were placed in 2-mm-diam plastic tubes which were inserted into increment boring holes 5 mm in diameter. Two large lateral roots, four positions up the trunk, and two branches were monitored. From two to four glass rods were placed laterally in each monitoring position in holes bored to the center of each tissue. Thus, dose measurements were made in terms of lateral distribution of the radionuclide as well as vertical distribution from the roots to the crown.

Average values for gamma dose rate to tree components during the 1963 growing season are being ascertained. These changes in dose rate over time reflect some of the physiological developments associated with transfers of minerals in the tree. The glass rods were inserted (March 18) before the tree leafed out. By the time of the first reading (March 29), the buds had opened and small leaves were present. The integrated dose to trunk (2.9 rads) and branches (1.7 rads) during this period was lower than the integrated dose to roots (4.9 rads), indicating the presence of larger amounts of Cs^{137} which overwintered in the root system. As the leaves developed, a net movement of Cs^{137} out of the root system into trunk and branches was evidenced by decreasing dose rate in the former and increasing dose rate in the latter. This method of following radionuclide movement from one plant component to another is faster and easier than harvest-radioassay techniques. Also, it involves no destructive sampling of the material under investigation. Use of a radiosensitive tool, such as the glass-rod dosimeter, therefore appears to be a good method of establishing the time at which many transfers of radionuclides are made from one plant component to another.

Total doses delivered since March to tissue of this inoculated tree are below those believed necessary to produce visible morphological damage. The average integrated doses were 25.7 rads to roots, 59.9 rads to trunk, and 34.0 rads to branches. However, the developing seed of this tree will have accumulated an appreciable gamma dose by time of maturity. Moreover, much of this total dose will have been delivered during critical, radiosensitive stages of development. Plans are in progress to compare germination and survival percentages of these inoculated tree seeds with those of control seeds collected from noninoculated trees in the same area.

Uptake and Accumulation of Strontium and Calcium by Algae. — The uptake of strontium and calcium by algae was studied to determine the influence of stable strontium and calcium concentrations in the culture medium on the accumulation process. The concentrations of calcium and strontium in the media were varied within the limits normally found in freshwater environments. Strontium-85 and calcium-45 were used to tag the culture media, and algal samples were counted to determine the uptake of the two radionuclides. The uptake of stable strontium by the algae increased by a mean factor of 8.5 with each tenfold increase of strontium in the medium and decreased by a mean factor of 1.7 with each tenfold increase of calcium in the medium. The uptake of stable calcium increased by a factor of 4 with a tenfold increase from low to intermediate calcium

levels and increased by a factor of 10 with a tenfold increase from intermediate to high calcium levels. The concentrations of strontium in the media did not influence the uptake of calcium. Uptake of the Sr^{85} isotope did not vary with increases in the media concentrations of strontium; however, uptake of both Sr^{85} and Ca^{45} was significantly higher at the low calcium concentration than at the intermediate and high calcium concentrations. The results indicate that the uptake of strontium by algae is primarily a function of the concentration of strontium in the environment with a slight inverse influence by calcium concentrations.

Analysis of Some Radiation Hazards from a Specific Postattack Environment. — The magnitudes of the hazards were estimated for a hypothetical soil-plant-cow-milk-man food chain on the radioactive White Oak Lake bed. A primary assumption was that the radioactive soil and vegetation of White Oak Lake bed are a simulacrum of a postattack environment. Two secondary assumptions were that the lake bed was tilled and used for production of dairy and beef products, and that short-half-life radionuclides (physical half-life less than 1 yr) had decayed to negligible activity. The radionuclides remaining in significant activity were Sr^{90}, Cs^{137}, Co^{60}, and Ru^{106}.

There does not appear to be a serious health hazard to man from consumption of dairy products and beef of cattle grazing on vegetation from White Oak Lake bed. In the case of children consuming milk contaminated by Cs^{137}, an annual dose of 4 rems could be received, and this is in excess of the Radiation Protection Guide of 0.5 rem/yr recommended by the Federal Radiation Council. More data on the bovine physiology of Co^{60} and Ru^{106} are needed to refine the calculations, but it appears that any new data could only decrease the estimates of the hazards from ingestion of these nuclides, because the most conservative approach was taken.

Evaluation of Fission Product Distribution and Movement in White Oak Drainage System. — A series of 21 discharge measurements was made in White Oak Creek basin on July 12, 1963, during base flow conditions, with pygmy current meters.

During the measurements, discharge from White Oak Dam was 8.9 cfs. The purpose of the measurements was to locate in detail, and as accurately as possible, points or reaches of channel contributing significant quantities of water to White Oak Creek during base flow conditions, that is, while water flowing in the base originates primarily as groundwater discharge. In general, results of the measurements indicate that the Knox dolomite of Chestnut Ridge was contributing 13% of the total flow; and the combined discharge of the Knox, the Chickamauga limestone of Bethel Valley, and the effluent discharged by ORNL accounted for 88% of the total flow. Discharge from Melton Valley, which includes about 40% of the basin area, and which is underlain mostly by the Conasauga shale, accounted for the remaining 12% of White Oak Creek discharge to the Clinch River.

RADIOLOGICAL PHYSICS, HEALTH PHYSICS, AND INSTRUMENTATION

Internal Dose Estimation: Stable-Element Metabolism by Man. — The influence of age, race, and geographical location has been tested statistically on the trace-element concentration in aortas, kidneys, livers, and

lungs from persons of Caucasoid and Negroid races who were long-term residents of the United States, India, and Africa. On the whole, the concentrations of trace elements in children were not different from those in adults, but certain elements in certain tissues showed significant variations: (1) Calcium in the aorta was significantly lower ($p \leqq 0.01$) in American children than in American adults. (2) For each regional group, concentrations of cadmium in the kidney were significantly lower in children than in adults. (3) In the children from the United States, lead was consistently significantly lower than it was in adults; but in the samples from India and Africa, this element showed no significant difference between children and adults. Females from each location were compared with males of the same age range (20–59 yr) from the same location. Except for higher lead ($p \leqq 0.05$) in the kidney, liver, and lungs of males, no sex differences were found in the American group. The African females had significantly higher ($p \leqq 0.01$) cadmium in the kidney than the African males. The Indian males and females showed no significant differences.

There appeared to be no significant differences due to race *per se*. Except for higher lead in lung and kidney (which is probably a reflection of occupational exposure), the American Negroid group was not significantly different ($p \leqq 0.01$) from the American Caucasoid group in any respect. At this level of significance, the African Negroes differed from African Caucasians only in higher calcium in kidney and molybdenum in liver.

The American Negroes were significantly different ($p \leqq 0.01$) from the African Negroes for many elements, and the American Caucasians were significantly different ($p \leqq 0.01$) from the African Caucasoids for approximately the same elements. The African Caucasians and Negroes had more aluminum in aorta, kidney, and liver and less cadmium and lead in kidney, liver, and lung than did the similar racial groups in the United States. The Indian Caucasians did not differ from the African Negroes in as many respects nor as significantly as they did from the American Caucasians.

Internal Dose Estimation: Estimates of $(MPC)_W$ for Occupational Exposure to Sr^{90}, Sr^{89}, and Sr^{85}. – Human data were used to obtain $(MPC)_W$ values for continuous occupational exposure to Sr^{90}, Sr^{89}, and Sr^{85}. A three-exponential model was found to give an empirically adequate representation of the data on retention and excretion of strontium by grossly normal adults for a period of 200 days following intake. The third exponential was selected to be roughly consistent with the data on stable strontium in diet and in bone of adults, as well as in fair agreement with the estimates of bone turnover rates by Rowland. This biological retention is described by the formula

$$R(t) = 0.73\, e^{-0.693t/3} + 0.10\, e^{-0.693t/44} + 0.17\, e^{-0.693t/4000} .$$

Various authors have used a power function to describe retention of strontium, and this model and the exponential model were found to yield comparable chronic-body-burden values when adjusted to fit the data from the same adults. From these estimates of the body burden resulting from long-term exposure at a constant level, the daily intake, or equivalently the maximum permissible concentration in water, $(MPC)_W$, could be estimated.

The observed ratio, O.R., of strontium and calcium in diet and in bone also were used to obtain an $(MPC)_w$ for Sr^{90}. It was found that all these models indicate an $(MPC)_w$ for Sr^{90} which is higher by a factor of 4 or more than that currently recommended by ICRP and NCRP.

ISOTOPE DEVELOPMENT

Nuclear Analysis Research and Development: Activation Analysis in Nutritional Studies. — Twin lambs separated and raised on pastures of differing trace-element concentration exhibited different viability and health characteristics. Experiments performed in New Zealand indicate that the concentration of manganese in the liver, kidney, and pancreas of killed animals reflects the high or low concentration of this element in the soil. Activation analyses made at ORNL of wool from such animals indicate that the wool may be a more sensitive indicator of trace-element deficiency than the vital organs. This procedure has the additional and obvious advantage of allowing the animal to continue living and thus be available for further studies.

Nuclear Analysis Research and Development: Enforcement of Narcotics Laws. — The gamma-ray spectra obtained from the neutron activation of more than 100 opium samples from known sources have been plotted and cataloged with the aid of the IBM 7090 computer. A code to call forth spectra from the catalog which most closely match the gamma-ray spectrum of an unknown has been written; efforts to use it have proved only moderately successful, but planned changes should make the call-out more definite and restrictive.

Recovery of Fission Products. — By the use of silver-catalyzed ammonium persulfate as oxidant, cerium was oxidized and extracted effectively with di(2-ethylhexyl)phosphoric acid (D2EHPA) to give separation from other fission-product rare earths. The feed solution was the rare-earth strip product (1.2 M in free nitric acid) from the D2EHPA process for recovering strontium and mixed rare earths from Purex 1WW waste. The oxidation of cerium in the presence of the solvent phase was much more effective than oxidation prior to extraction. The cerium extraction coefficient (1/1 phase ratio) in 1 hr of contact time at 50 to 55°C was about 25.

Of several stripping agents examined for recovering technetium from Aliquat 336, a quaternary amine extractant, only perchloric acid (2 to 6 M) gave stripping coefficients greater than 1.

Curium-242 Preparation: Recovery: Control of Acidity in High-Activity-Level Tramex Feed. — The sensitivity of separations by the Tramex process to small changes in acidity makes control of acid losses through radiolysis exceptionally important. Studies of this problem are continuing.

Preliminary investigations were made to determine whether hydrazine monohydrochloride or formaldehyde added to high-activity-level Tramex feed would stabilize the acid at a concentration in the 0.01 to 0.1 N range, which is necessary for satisfactory extraction. When $N_2H_4 \cdot HCl$ was

present in high-activity-level Tramex feed, the G value for H^+ production was about 1.4. In the absence of $N_2H_4 \cdot HCl$ the G value for H^+ destruction is about 1.4. This G value for H^+ production is equivalent to an acid increase of about 0.1 mole per liter per day for a Cm^{242} power level of 10 w/liter. Presumably the hydrazine reacts to convert chlorine in radiolysis products to HCl and at the same time releases HCl chemically bound in the $N_2H_4 \cdot HCl$ salt.

When 1% formaldehyde was present in 10 M LiCl containing 10 w of Cm^{242} per liter, the H^+ increased only 0.066 mole/liter in seven days. This is equivalent to an acid-production G value of about 0.14. These results indicate that acid concentration in Tramex feed can be satisfactorily controlled by the addition of small amounts of formaldehyde. Additional study is needed to confirm the validity of these conclusions.

Curium-242 Preparation: Full-Activity-Level Tramex Flowsheet Test. — A total of five hot runs have been made at Cm^{242} power levels of 5 to 10 w/liter. Operating difficulties were slight except for the last run, in which the air lift pump for hot feed became inoperable. Although none of the runs was made at proper conditions for both high curium recovery and complete fission product decontamination, the results indicate that the Tramex process should be capable of producing a high yield of acceptable product.

No effect of radioactivity was noted on the solvent extraction operation at 10 w/liter; however, radiolysis of the low-acid feed did cause precipitation at some time between 28 and 40 hr after feed adjustment.

Curium-242 Preparation: Effect of Nitrate on Extraction in the Tramex Process. — The addition of small amounts of Alamine $336 \cdot HNO_3$ to Alamine $336 \cdot HCl$—diethylbenzene is being investigated as a possible means of increasing actinide distribution coefficients. With higher distribution coefficients, control of Tramex feed acid concentration should be less critical.

Distribution coefficients between 0.6 M Alamine $336 \cdot HCl$—diethylbenzene and 10 M LiCl—0.15 M $AlCl_3$ were 6 for americium and 0.05 for europium. When the extractant was 0.5 M Alamine $336 \cdot HCl$—0.1 M Alamine $336 \cdot HNO_3$—diethylbenzene, distribution coefficients were increased to 22 for americium and 1.2 for europium. The effect of free acid in this system is being investigated.

Curium-242 Preparation: Laboratory Demonstration of Chloride-to-Nitrate Conversion for Curium-242 Product. — Curium-242 product from the Tramex process will be contained in approximately 12 liters of 8 M HCl. The proposed method of converting this to a nitrate solution is to evaporate to 4 liters, add 4 liters of concentrated nitric acid, and evaporate to 4 liters. This process is to be repeated two more times.

This conversion method was tested at 1/10 scale in tantalum equipment with simulated feed containing mixed rare earths as substitutes for americium and curium.

The chloride concentration of the product was reduced to 1 or 2 ppm after the second nitric acid boil-off and to less than 1 ppm after a third boil-off. The final product was approximately 14 M HNO_3 containing 0.5 g of rare earths per liter. Noncondensable acid reaction products (probably nitrosyl chloride) were readily scrubbed from the off-gas with dilute caustic. From all three boildowns a total of about 5.5 equivalents of acid were collected in the caustic trap for each liter of product.

Ruthenium added as Ru^{3+} chloride did not volatilize during the conversion to nitrate. Methods to promote ruthenium volatilization are being investigated.

Radioisotope Research and Development. — Cesium-137 aluminosilicate glass was prepared by the absorption of cesium ion on finely divided Decalso (synthetic sodium aluminosilicate exchanger). Leach rates on the glass were 1.1 $\mu g\ cm^{-2}\ day^{-1}$ in tap water at 20°C. (The leach rates on previously reported cesium borosilicate glasses were ~1000 times as high.) This procedure for preparing cesium glass eliminates the need for grinding and blending operations and also reduces the amount of Cs^{137} volatilized during the glass preparation steps.

A series of iridium irradiation experiments have been conducted in the ORR to produce Ir^{192}. Data from these experiments determined a neutron cross section for Ir^{192} of 1220 ± 280 barns.

A study of the use of the fast-neutron flux of reactors was initiated. Several targets of iron were inserted in the LITR and ORR isotope facilities for study of the $Fe^{54}(n,p)Mn^{54}$ reaction. A partial fuel element for the LITR was used in addition to the standard loading points. Processing of these targets is in progress. The LITR partial fuel element was loaded with 60 mg of 92.4%-enriched $Zn^{67}O$ for the $Zn^{67}(n,p)Cu^{67}$ reaction. A yield of 180 μc of Cu^{67} was obtained for a saturation irradiation. A similar irradiation performed in the hydraulic tube of the ORR for a 133-hr irradiation produced ~25 μc of Cu^{67} from a 12.4-mg sample of 92.4% $Zn^{67}O$. These results, when corrected for various factors and scaled up by a factor of 10 (as an approximation of the difference in flux between the LITR and the ORR) indicate that the production of Cu^{67} in a partial fuel element of the ORR would be ~20 times that produced in the hydraulic tube facility of the ORR.

Radioisotope and Stable Isotope Production. — Ion currents equivalent to 2875 g of separated isotopes were monitored during the two-month interval. Isotopes of Ca, Si, Ba, S, Cd, Rb, Cr, and Pb were separated and collected using 28 of the calutrons associated with the program.

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