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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

MARCH 1963

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Oak Ridge, Tennessee
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UNION CARBIDE CORPORATION
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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

MARCH 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 PROGRAM

GAS-COOLED REACTOR PROGRAM

GCR-ORR Loop No. 1. — Two ORR cycles of operation with a fuel element having a roughened surface were completed, and the element was removed for postirradiation examination. A new roughened-surface element having a higher heat rating, estimated at 65,000 Btu hr^{-1} ft^{-1}, was installed. This assembly is equipped with pressure-measuring taps and transducers for determining the pressure drop over the roughened-surface element. Preliminary data indicate the performance of the element to be approximately that expected.

During the reactor shutdown, a smear test was made in the tee section through which fuel elements are removed, to measure the radioactivity of the dust in the loop. A direct reading of 30 mr/hr was obtained, and all the activity was identified as components of stainless steel. The set of two compressors in the loop continues to operate satisfactorily.

GCR-ORR Loop No. 2. — Loop No. 2 operated satisfactorily to the end of the reactor cycle. Traces of fission products from the UO_2-coated fuel element in the in-pile portion of the loop were found in large gas samples removed from the helium circuit. The low-power flux calibration test is being continued into the next reactor cycle. Enough fabricated parts for the fuel holder assembly were received to initiate construction.

LITR Capsule Irradiations. — The capsule containing pyrolytic-carbon-coated fuel particles manufactured by General Atomic continues to operate satisfactorily after having achieved a calculated burnup of 4.5 at. % of the heavy metal. Temperatures inside the a and b fuel compartments are, respectively, 1680 and 1900°F, compared with 1935 and 2115°F at the start of irradiation in June 1962.

ORR Capsule Irradiations. — The sweep capsule containing 1-1/2-in.-diam balls with UC_2-coated particles uniformly distributed in the matrix (manufactured by National Carbon Company) continues to operate without

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detectable release of fission gases. This capsule, which has been irradiated two months at approximately 1500°F, has an estimated burnup of 1.7 at. % of the heavy metal.

ETR Capsule Irradiations. — Capsule E-8, having an estimated burnup of approximately 7000 Mwd per metric ton of UO_2, was discharged from the reactor and delivered to the hot cells for examination. This capsule contained hollow UO_2 pellets and operated at a maximum cladding-surface temperature of 1600°F. Ten capsules remain in the ETR for additional irradiation.

Instantaneous Fission-Gas Release from UO_2. — Large specimens of thin-plate single-crystal UO_2 are being irradiated to study the basic mechanisms of fission-gas release. The effect of changing irradiation conditions on krypton release was observed for these specimens. The specimen temperature was cycled by rapidly cooling and reheating to the original temperature. This process did not produce bursts of fission gas and demonstrated that thermal stresses do not cause the heating burst.

Heating bursts were found to release gas in the same isotopic composition as the steady-state release; this implies that the burst is an accelerated version of the steady-state release. The amount of krypton in the burst is more than that predicted by a change of diffusion coefficients; this implies that neither steady-state nor burst release occurs by a simple diffusion process. The krypton (Kr^{85m}, Kr^{87}, and Kr^{88}) release rates all reach equilibrium within 3 hr after irradiation begins; however, diffusion theory indicates that equilibrium release of these isotopes should require at least 18 hr for Kr^{85m}, 11 hr for Kr^{88}, and 5 hr for Kr^{87}. The burst mechanism affects fission-gas release for only about 3.6 hr; thus it cannot totally account for the quick equilibrium. The rapid attainment of equilibrium also implies that simple diffusion is not the release mechanism for the fission gas at temperatures below 1000°C.

Irradiation Testing of Stainless Steels. — Out-of-pile stress-rupture tests on a 20% Cr—25% Ni—1% Nb—bal Fe alloy at 750°C in an atmosphere of CO_2—5% CO indicate no difference in stress-rupture properties compared with those obtained during tests in argon containing 1% O_2. In-pile stress-rupture test results suggest a slight weakening at 650°C and no effect at 750°C. Strain-at-fracture results for these tests are lower than corresponding out-of-pile test results by 30 to 50%. A number of the in-pile specimens failed in or near the end-closure welds, indicating possible irradiation-induced weakening or embrittlement at the welds, or both.

Coated-Particle Inspection Techniques. — Preliminary tests with CCl_4 as an opaque impregnant for use in the radiographic detection of cracks and other flaws in particle coatings indicated sufficient improvement to justify additional tests.

Advanced Fuel-Element Mass-Transfer and Pressure-Drop Experiment. — Pressure-loss coefficients were determined in a Reynolds-modulus range from 2×10^3 to 2×10^5 for bare rods and for rods spirally wrapped with 0.006-in.-diam wire on a 0.036-in. pitch (pitch-to-height ratio = 6:1).

At low Reynolds moduli, approaching laminar flow, both configurations exhibited similar pressure-drop characteristics; at the higher Reynolds moduli, the roughened surface produced greater loss coefficients. For $N_{Re} > 10^5$, the pressure-loss coefficients of the wire-wrapped surface are approximately twice those of the smooth configuration.

Because of difficulties in installing and removing the cluster from the channel (necessitating appreciable time delays), no satisfactory mass-transfer data were obtained for the bare-rod configuration. Since allowable exposure times are much less for the wire-wrapped rods (mass removal causes the wire coil to collapse), it is not anticipated that satisfactory mass-transfer data will be obtained without extensive modification of the apparatus. Accordingly, this phase of the experiment was abandoned.

Release of Fission Products by In-Pile Burning of Uranium Carbide–Graphite Fuels. – Three irradiation experiments were conducted in the ORR to study the release of fission products that would occur in a reactor accident in which air was admixed with the helium coolant. In these in-pile experiments, a uranium carbide–graphite fuel specimen was exposed to air during high-temperature operation in the ORR and partially burned. The fuel specimen consisted of pyrolytic-carbon-coated uranium carbide particles in a graphite matrix made by Minnesota Mining and Manufacturing Company. Approximately half the specimen was consumed in the 15-min period of burning, during which the temperature reached 1400°C.

Radiochemical analyses showed that if combustion of graphite-uranium fuel occurred in a reactor accident under the conditions of these experiments, all but a small fraction of the strontium, zirconium, barium, cerium, and uranium would remain in the vicinity of the fuel and that 15 to 25% of the iodine and tellurium and 35 to 40% of the cesium would be released. The release of ruthenium would vary from 5 to 40%. The association of a disproportionate amount of the uranium with the unburned fuel indicates that the part of the specimen with the least uranium burns preferentially.

Gaseous Diffusion in Porous Media. – The "dusty-gas" model developed to correctly account for the mutual isothermal diffusion of gases through porous media at a uniform pressure was extended to include the case of pure-gas flow under nonisothermal, nonuniform-pressure conditions. The resulting equations are strikingly similar in form to previous empirical and semiempirical equations used to correlate thermal transpiration data.

AVR Physics. – A survey was made of computational methods for the effective resonance integral of thorium in the AVR (German pebble-bed reactor). The situation is complicated because two types of heterogeneities exist in the fuel: the coated particles are distributed within a ball in a random fashion, and the balls are distributed within the core in a random fashion. It was concluded that both types of heterogeneities could most satisfactorily be taken care of by an equivalence relationship of the type suggested by Lane, Nordheim, and Sampson [Nucl. Sci. Eng. 14, 390 (1962)]. The Doppler-broadened resonance integral was calculated by using the infinite-mass approximation in the first, second, and fourth resonances and the narrow-resonance approximation in all other resonances.

The calculation of the control-rod worth for the AVR was started. Control-rod boundary conditions are being determined by one-dimensional

multigroup calculations. The control-rod worth will then be computed by two-dimensional few-group calculations for several different core compositions.

STUDIES AND EVALUATIONS

Reactor Evaluation Studies. – Modifications to the Expire reactivity lifetime code have been completed, and the revised code (renamed WIPEOUT) is being checked against experiments and the GAM-THERMOS calculational results. The WIPEOUT code allows fuel, coolant, and structural materials to be distributed in any desired order in up to 17 concentric annular regions. Thermal-group cross sections are calculated by the Wilkins heavy-gas model, using the Westcott parameters in a series of equations of the Breit-Wigner form to specify the non-1/v cross sections as a function of energy in the thermal range. Thermal-flux distributions are calculated by a one-group P-3 approximation to the Boltzmann transport equation. The criticality calculation and time-dependent isotope equations are the same as used in Expire.

The review of basic cross-section data (with special interest in the latest measurements on resonance cross sections) is almost completed. Every effort is being made to ensure that a consistent set of data reflecting the latest measurements is incorporated into the WIPEOUT library.

In order to expedite the parametric studies for the heavy-water-moderated reactors for desalinization applications, a subroutine to the WIPEOUT code has been written; the subroutine will automatically optimize concentric-tube fuel elements with respect to initial cell-power distribution (as calculated by the P-3 routine) and heat-transfer and fluid-flow requirements. With this feature incorporated into the reactivity lifetime, the code will automatically optimize the fuel element to satisfy engineering requirements and will then evaluate the reactivity performance and fuel-cycle cost resulting from this design.

An IBM 7090 code (DWNP) for water desalinization calculations is being developed. The code will compute optimum performance ratios and will calculate the costs of producing fresh water in evaporator plants using low-pressure steam exhausted from turbines generating electrical power with steam produced by nuclear reactors. In its present state of development the code uses, as input, estimates of capital costs of the reactor and turbine plants and of the fuel fabrication and processing plants, annual operating expenses, fuel exposure, thermal efficiencies, plant utilization factors, interest and amortization rates, etc., and calculates heat costs as parameters of the price of electricity. The evaporator performance ratio is then optimized from the calculated heat cost by means of an equation which relates heat cost and pumping requirements to performance ratio.

A preliminary study was made of the uranium utilization which might be achieved in a heavy-water reactor utilizing natural uranium as the feed material. A CANDU reactor employing bidirectional fueling was taken as the reference design. Reactivity lifetimes were calculated for various assumed fission product absorptions, with and without recycle of the by-product plutonium. It was found that with all fission product losses included and with no plutonium recycle reactivity, lifetimes of 9800 Mwd per metric ton can be achieved; subsequent use of the plutonium in a burner reactor would

add 3200 Mwd per metric ton, bringing the total energy yield to 13,000 Mwd per metric ton, or 1.3% effective utilization of the natural uranium. Elimination of all fission product losses, combined with recycle of the recovered plutonium with natural uranium, was found to increase the natural-uranium utilization to 2.4%.

Sargent and Lundy's estimates of the capital costs of large D_2O-moderated pressure-tube reactors were completed. Costs of 3500-, 8333-, and 25,000-Mw (thermal) plants were found to be $23.14, $16.14, and $13.03/kw (thermal) respectively.

REACTOR FUELS AND MATERIALS DEVELOPMENT

Nondestructive Test Development. – Application of x- and gamma-ray attenuation to determinations of inhomogeneity in reactor fuel plates and rods has brought about the design and fabrication of a versatile scanning apparatus. This machine employs a commercial 150-kv (constant pressure) x-ray unit as the radiation source for inspection of thin fuel plates and a 2-curie Co^{60} source for inspection of fuel rods or other heavy sections. The radiation is detected with an NaI(Tl) crystal optically coupled to a photomultiplier tube.

Fuel plates up to 50 in. long with core widths up to 5.75 in. can be inspected manually or automatically at any linear speed between 0 and 200 in./min. Indexing across the plate width can be accomplished manually or automatically in steps from 0.0005 to 5.75 in. Fuel rods up to 50 in. long can be inspected at the aforementioned linear speeds and step-rotated in increments of 1.8° or helically scanned with any desired pitch.

Since the output signal of the photomultiplier is representative of the fuel inhomogeneity, it is used to drive a strip-chart recorder and/or a computer for the total signal integration over a scan line.

This unit will be applicable for use during development of fuel plates and rods for projects such as the HFIR, ATR, BNL Kilorod, and Fuel Cycle.

Zirconium Metallurgy. – Single crystals of iodide zirconium up to 3/4 in. wide by 3 in. long and of Zircaloy-2 up to 1/4 in. wide by 2 in. long have been grown for the first time. In the newly developed technique, localized electron-beam heating is used to produce an extremely steep temperature gradient which is moved at a rate of 1/2 to 1 in./hr along a strip specimen 1/16 to 1/8 in. thick and 1/2 to 1 in. wide. At present, a transformation front (β phase to α phase) is passed along the specimen. Preliminary experiments indicate that the movement of a recrystallization front (cold-worked to annealed material) can also be used to produce the large grains, but at a slower rate. The maximum length of single crystal which can be produced seems to be limited only by the length of strip material which can be moved past the electron gun. The technique should be applicable to other zirconium alloys and to other materials.

Solid Reaction Studies. – Diffusion of Nb^{95} in pressed and sintered and in fused UO_2 was studied over the range 1100 to 2100°C. In every case the activity profiles (plots of ln activity vs distance squared) were characterized by three segments: an initial portion having a large Nb^{95} activity gradient that was slightly concave upward; an abrupt, smoothly

curved portion beginning at depths of 5 to 25 μ; a linear portion extending to the limit of Nb^{95} penetration. A large fraction of the total activity was contained in the region near the surface. Diffusion coefficients were calculated from the linear portion of the penetration plots for pressed and sintered UO_2 heat-treated at 1265 to 1600°C. These coefficients vary from 1.7×10^{-11} to 1.4×10^{-8} cm^2/sec. An Arrhenius-type plot of these values yielded an activation energy of 116 kcal/mole. Above 1600°C, however, the diffusion coefficients were approximately constant at 2×10^{-8} cm^2/sec. Although only three fused UO_2 specimens have been analyzed, the calculated diffusion coefficients give a linear Arrhenius-type curve. The values vary from 2.5×10^{-11} cm^2/sec at 1100°C to 2.0×10^{-9} cm^2/sec at 2010°C and yield an activation energy of only 30.1 kcal/mole. Grain-boundary diffusion may have been partly responsible for the shape of the activity profiles observed in pressed and sintered UO_2, since the grains were small, averaging about 30 μ in diameter; however, similar activity profiles were found for fused UO_2 in which the grains were several millimeters in diameter.

Materials Compatibility Studies. – Compatibility studies with the lithium-niobium system continued. The effect of prior deformation as a variable in the corrosion process was studied with single crystals having the same orientation but with different dislocation densities. A maximum penetration of 13 mils occurred in an annealed crystal containing 1700 ppm oxygen, as compared with 17 mils in a similar specimen which had been cold-rolled to a 20% reduction in thickness. In an earlier experiment (ORNL-3368, p 8) the deeper penetration occurred in the annealed specimen; consequently, it is not apparent at this time whether a relation exists between deformation and corrosion. In further tests, deformation will be followed by a low-temperature heat treatment in order to increase the oxygen diffusion rate without eliminating many dislocations.

The effect of grain size on mode and depth of lithium penetration into niobium was also investigated. At 400°C the depth of penetration increased with increasing grain size and the mode changed from primarily intergranular to transcrystalline. At 1000°C, penetration occurred along grain boundaries for all grain sizes and the depth of attack was greater than for the corresponding grain size at 400°C. It was concluded, therefore, that lithium penetration is influenced by both grain size and test temperature.

The addition of zirconium to niobium alters its corrosion behavior in lithium if any oxygen present in the alloy is in some way associated with the zirconium. It was found that after the addition of approximately 1000 ppm oxygen to Nb–1% Zr alloy at 1000°C, between 5 and 8 hr of further heat treatment at 1000°C is required to stabilize the alloy with respect to lithium. In tests conducted in 500°C lithium, 5 to 20 mils of attack was observed in specimens heat treated for less than 5 hr at 1000°C following contamination with oxygen, but no attack was observed in specimens heat treated for 8 hr or longer.

Fuel Element Development. – The effects of temperature and pressure on phase relations in the uranium-oxygen system are of interest in connection with fuel migration and loss and with coolant compatibilities in BeO-UO_2 fuel components. Accordingly, a study of the phase relationships in the uranium-oxygen system was initiated. A thermogravimetric apparatus was designed and constructed for use at temperatures as high as 1800 to 2000°C in vacuum or as high as 1400 to 1600°C in various gases. Preliminary

results show that above 1100 to 1200°C at 10^{-5} to 10^{-6} mm Hg, UO_{2+x} ($0 < X < 0.2$) loses weight, and the magnitude of this loss indicates that a volatile uranium species is present. Experiments at 1500 to 1800°C in vacuum showed that a volatile material with a composite oxygen-to-uranium ratio of 4 is produced during the reduction of the O/U ratio of UO_{2+x} to the equilibrium value of approximately 2.000. Experiments with a variety of uranium dioxides with varying O/U ratios all gave similar results. These data suggest that gaseous UO_4 is responsible for at least a portion of the uranium volatilized from UO_{2+x}, although the possibility of a volatile component consisting of $UO_3 + 1/2\ O_2$ cannot be excluded.

REACTOR COMPONENTS DEVELOPMENT

Reactor Controls Development: Reactor-Control Electronic Instruments. — A system of reactor-control modules has been devised; each member is a functional unit of one or more transistor electronic instruments for reactor use. The magnet-control amplifier, pulse amplifier, and low-current dc operational amplifier previously described have been converted to modular form, along with other circuits developed or under development. In all, 24 modules are presently envisioned, and the scheme is capable of almost indefinite expansion. The outside dimensions of the assemblies are compatible with presently developing international standards for reactor-control instruments.

Circuits recently developed include a "logical-or," to combine the several inputs to a safety system, and a compact ionization-chamber high-voltage supply, with monitoring and testing circuits.

The wide-range counting channel [Trans. Am. Nucl. Soc. 3, 454 (1960)] has been under test in the ORR for several weeks. Preliminary results, after the instrument followed several reactor shutdowns and startups, are very encouraging. The instrument easily follows the power of the ORR over its entire range without any switching. Its two output signals are the reactor period and the logarithm of the reactor power.

Reactor Controls Development: Use of Gamma Radiation as a Reactor-Control and Safety Parameter. — Several experiments with gamma-sensitive ionization chambers in the BSR and the ORR have led to an improved understanding of the advantages and limitations of using gamma radiation with respect to the neutron flux usually used. The principal advantage is that the effect of changes in core and shielding configurations, at least in water-shielded reactors, is greatly reduced. The disadvantages are the fission product component of the signal, (20 ± 1)% at equilibrium in the BSR, which is related to the prior history of the reactor, and the signal from capture gamma rays produced by neutrons far from the core. The latter effect was observed whenever the neutron-to-gamma flux ratio exceeded 0.2 nv per photon cm^{-2} sec^{-1} anywhere along the path between the core and the detector. Older experiments at SPERT (ORNL-3393) at periods as short as 3 msec confirmed, as expected, that gamma-sensitive chambers would work well as safety detectors. The results of these experiments and several years' operating experience with gamma chambers in the ORR and the BSR lead to the conclusion that the use of gamma radiation for reactor control and safety is feasible, and sometimes advantageous.

POWER REACTOR FUEL PROCESSING

Shear-Leach Process. – Bench-scale leaching tests on sheared ThO_2-UO_2 fuel were continued. Leaching of 1/2-in.-long sheared lengths of stainless-steel-clad ThO_2-UO_2 in a 4-in.-diam cylindrical dissolver resulted in the dissolution of 85% of the ThO_2-UO_2 in 2 hr in recirculating 12 M HNO_3–0.10 M Al–0.04 M NaF. The high dissolution rate demonstrated the effectiveness of an air-operated, adjustable draft tube in keeping the ThO_2-UO_2 fines in suspension. In comparison, two subsequent dissolution runs reached 85% dissolution in about 5 hr, apparently because of the poorer suspension produced by a fixed draft tube. Suspension of ThO_2-UO_2 fines during dissolution remains a major problem.

Processing of Breeder Reactor Fuels. – Studies on the kinetics of UO_2 pellet dissolution were continued. In 14 M HNO_3 the instantaneous dissolution rate of UO_2 pellets having the same density (10.3 g/cm^3) varied, depending upon the manufacturer; however, the times for complete dissolution of all pellets 1 cm long by 1 cm in diameter were about the same. The variation in apparent surface area during the dissolution compensated for the difference in initial rate (15 to 330 mg cm^{-2} min^{-1}), and the time for 90% dissolution varied only from 60 to 83 min.

Uranium Dicarbide Metallographic Studies. – Heating of $UC_{2.00}$ or $UC_{1.91}$ (both with a combined-C/U ratio of 1.86) for 60 hr at 1450°C had no effect on the combined-C/U atom ratio, x-ray powder pattern, or amounts of gaseous hydrocarbons produced upon hydrolysis. The as-cast $UO_{2.00}$ was a two-phase mixture of $UC_{1.86}$ and graphite, which appeared as rod-shaped areas in the photomicrographs. After heating, small platelets of a third phase were visible at 1000×. Similarly, as-cast $UC_{1.91}$, which appeared to be nearly single-phase, contained the same small platelets after heating. These platelets have been observed in all dicarbide specimens after heat treating at temperatures between 1260 and 2000°C. No thermal decomposition of $UC_{1.86}$ to U_2C_3 and free carbon was detected at any of the temperatures where heat treatment was conducted. These results are in disagreement with those obtained at Battelle Memorial Institute, where the decomposition was reportedly observed at temperatures below 1500°C.

Hydrolysis of Thorium Carbide. – The maximum amount of carbon that could be combined with thorium by arc melting (using graphite electrodes) was 9.2 wt %, which corresponds to $ThC_{1.95}$. Therefore, thorium dicarbide, like uranium dicarbide, is carbon deficient, although $ThC_{1.95}$ is much closer to the idealized MC_2 formula than is $UC_{1.85}$.

Reaction of thorium dicarbide samples (total-C/Th atom ratios of 1.95 and 2.14 and a combined-C/Th ratio of 1.95 in both cases) in water at 80°C yielded 49 ml (STP) of gas per gram of carbide. The evolved gas consisted (by volume) of 30% hydrogen, 3% methane, 32% ethane, 5% C_3-to-C_8 alkanes, 12% alkenes, 14% alkynes, and 2% unidentified unsaturates. This gas accounted for 50% of the combined carbon; an additional 12% was found as a water-insoluble, ether-soluble wax. The remaining 35% of the carbon could not be accounted for and presumably was soluble in the aqueous phase, which had a strong organic odor.

The thorium dicarbide hydrolysis products were in general similar to those from uranium dicarbide. The amounts of the various alkanes and

alkenes produced in the hydrolysis of $ThC_{1.95}$ were nearly identical with those obtained by extrapolating the hydrolysis data on as-cast uranium carbides to a hypothetical composition of $UC_{1.95}$. In addition, thorium dicarbide yielded significant quantities of alkynes, which accounted for 14% of the combined carbon and were principally acetylene and butyne-1. The alkyne content of the off-gas from $UC_{1.85}$ hydrolysis was only 1%, and no acetylene was observed. This accounts for the fact that the total carbon in the gas phase was 50% for $ThC_{1.95}$ vs 35% for hypothetical $UC_{1.95}$. Thorium dicarbide yielded less wax, 12% vs 25% for uranium dicarbide, and about the same amount of unaccounted-for, soluble organic compounds.

Thorium dicarbide, like uranium dicarbide, contains C_2 units in the crystal lattice, which yield predominantly C_2 compounds or multiples-of-C_2 compounds upon hydrolysis. Hydrolysis of $ThC_{1.95}$, which approaches MC_2, produced only 1% methane, in keeping with the hypothesis that methane is the hydrolysis product of only single C units in the crystal lattice and that the C—C bonds in C_2 units are not broken during hydrolysis. The thorium dicarbide structure is not exactly the same as that of uranium dicarbide, so some variation in hydrolysis products might be expected.

Chlorination of UC_2-ThC_2-Graphite Fuels. — Three 10-g samples of UC_2-ThC_2-graphite fuel containing 1.5% uranium and 7% thorium were burned in oxygen at 800°C in 2.5 to 3.5 hr. The product ash was then chlorinated for 2 hr with 15% CCl_4—Cl_2 at 500 and 550°C for two of the samples, and with phosgene at 500°C for the third sample. Complete chlorination was achieved, and the unvolatilized products were completely soluble in water in all three tests. The respective amounts of uranium volatilized from the residues during chlorination were 74, 84, and 64% of the total present. The volatilized uranium chlorides were collected on the cool exit of the reaction tube.

These experiments indicate that it is possible to process this type of fuel in a nickel vessel to yield an aqueous solution of the product, containing both the uranium and the thorium. Quantitative separation of uranium and thorium may be possible by using higher temperatures to completely volatilize the uranium as uranium chloride, but ceramic vessels would have to be used, because metal vessels would corrode too rapidly at the temperatures required.

Hydrochlorination of U-Al Alloy Fuel. — The maximum steady-state temperature during hydrochlorination of an MTR fuel element, as calculated with the GHTR heat transfer code on the IBM 7090 computer, was found to decrease from 1073°C at a hydrochlorination rate of 12 mils/hr to 549°C at 6 mils/hr. At the average rate of 6 mils/hr, the 60-mil-thick inside plates and the 180-mil side plates could be completely hydrochlorinated in 5 and 15 hr respectively. The additional small contribution to heat removal by the heat capacity and convection of gases present was not considered. These results indicate that it will be possible to hydrochlorinate MTR fuel elements at rather low rates without exceeding the melting temperature of the fuel, 650°C.

An experiment showed that the volatile-uranium loss during hydrochlorination of an 8% U-Al alloy could be held to 0.5%. The experiment was designed only to investigate the beneficial effect of using carbon monoxide as a carrier gas. Mixed chlorides of uranium and aluminum were prepared by chlorinating and volatilizing the U-Al alloy and then condensing

and collecting the volatilized chlorides at room temperature. Three runs were then made by passing argon at 300°C, carbon monoxide at 300°C, and carbon monoxide at 200°C over separate samples of the mixed uranium and aluminum chlorides in order to selectively volatilize the aluminum chloride. The percentages of uranium lost by volatilization with the aluminum were 18.3, 8.3, and 0.5 respectively. Slightly volatile fractions collected in the hot zone contained 59.7, 2.7, and 97.9% of the uranium respectively. This fraction also contained from 2 to 4% of the aluminum in all three cases. An additional 22.0, 88.9, and 1.6% of the uranium, respectively, was found in the nonvolatile fraction from each run, along with 3 to 6% of the aluminum.

It is apparent that the use of the higher temperature of 300°C with argon, and particularly with carbon monoxide, helped keep uranium in the nonvolatile fraction, while the use of the lower temperature of 200°C with carbon monoxide caused the volatilized uranium to redeposit almost quantitatively within the hot zone, thereby greatly decreasing the amount of uranium lost by volatilization with the aluminum. Therefore, a separation procedure is suggested, based on the selective condensation of uranium chloride from a vapor of the mixed chlorides in a chamber, the entrance and exit ends of which are heated to about 300 to 200°C, respectively, using carbon monoxide as a carrier gas.

The beneficial effect of carbon monoxide in these experiments can be explained by its ability to reduce volatile uranium chlorides in the gas phase to lower-valent, nonvolatile compounds. The loss of uranium by volatilization is a serious problem in the hydrochlorination of U-Al alloys, because the $AlCl_3$ chlorination product is capable of oxidizing much of the normally expected uranium chloride (UCl_3) to higher-valent, volatile chlorides.

Corrosion. — Two sets of corrosion experiments were made in order to evaluate various materials of construction for chloride volatility processes. Haynes alloy No. 25, Nichrome V, INOR-8, nickel, and type 304L stainless steel corroded at maximum rates of 0.02, 0.08, 0.01, 0.05, and 0.12 mil/month, respectively, for 264-hr exposures in hexachloropropene or its vapors at 185 to 190°C. Rates for niobium and tantalum for similar exposures of 168 hr were 0.95 and 0.05 mil/month respectively. In oxidation-chlorination cyclic tests consisting of 5 hr in O_2 at 800°C followed by 5 hr in Cl_2-CCl_4 at 500°C, rates for five cycles of exposure with INOR-8, Nichrome V, and "A" nickel were 24, 7, and 754 mils/month respectively. Specimens exposed during cycling in the development of the process on a laboratory scale did not show such high rates.

Protactinium Adsorption. — Studies on the adsorption of protactinium on unfired Vycor were continued. Protactinium-233 was decontaminated from uranium by a factor of 1.59×10^4, using a column 10 mm in diameter and 8.6 cm long, containing 5 g of 60- to 80-mesh unfired Vycor. The feed solution contained uranium, 11.2 mg/ml; thorium, 9 mg/ml; fluoride, 0.04 M; aluminum, 0.1 M. It was 10 M in HNO_3, and the Pa^{233} concentration was equivalent to 2.04×10^6 counts min^{-1} ml^{-1}. The solution flow rate was 0.64 ml min^{-1} cm^{-2}. After the loaded column was washed with 28 ml of 10 M HNO_3, about 80% of the protactinium was recovered by eluting with 18 ml of 0.5 M oxalic acid. The uranium in the combined raffinate and wash was decontaminated from the protactinium by a factor of 44.

The corrosion of the unfired Vycor by the fluoride in the feed solutions was decreased by more than a factor of 50 by the presence of 2.5 moles of aluminum per mole of fluoride ion. Solutions 0.05 M in HF and 11 M in HNO_3, with and without 0.12 M Al, were passed through columns 4 mm in diameter and 9 cm long containing 1 g of 60- to 80-mesh unfired Vycor at a flow rate of about 1 ml min^{-1} cm^{-2}. After about 200 ml had passed through the columns, the emerging raffinate from the aluminum feed solution contained silicon to the extent of 0.006 mg/ml, whereas the solution without the aluminum contained 0.32 mg/ml.

Stripping of Uranium from 2.5% Di-sec-butylphenyl Phosphonate (DSBPP) in Diethylbenzene (DEB). – The discrepancy between laboratory, Unit Operations, and pilot-plant data of the stripping of uranium from 2.5% DSBPP in DEB is apparently due to degradation products in the solvent. Used solvent from the pilot plant was equilibrated with a feed solution containing U^{233} and then exhaustively stripped with one-quarter volume of water. The stripping coefficient decreased from about 200 with the first contact to a constant 0.5 after the fifth contact. This indicated that about 1% of the uranium (5×10^4 counts min^{-1} ml^{-1}) was strongly complexed by the solvent, probably due to the presence of organic degradation products.

Solvent Extraction of UC Fuel Solutions. – The UC in sheared, stainless-steel-clad uranium monocarbide (Consumer's Public Power District Reactor, core 2) reactor fuel may be converted to uranyl nitrate–nitric acid solution for Purex solvent extraction either by hydrolysis with water at 80 to 100°C to yield hydrous UO_2, followed by dissolution in nitric acid, or by direct dissolution with nitric acid. The first alternative head-end procedure has been coupled to a conventional Purex process and is being cold-tested.

Hydrolysis of 30-g specimens of impure UC containing 5.1% C with water at an H_2O/UC mole ratio of 20, at 90°C for 4 hr, yielded a thick slurry of hydrous UO_2. Dissolution of the slurry in boiling 8 M HNO_3 for 2 hr yielded an orange-colored, raw feed solution that was 2 M in HNO_3, with a uranium concentration of 300 g/liter. This feed solution, in contrast to more dilute feeds prepared by dissolution in 4 M HNO_3, was free of waxy solids. The concentrated raw feed contained traces of soluble organic acids that resulted from hydrolysis of the dicarbide impurity in the fuel. Unless removed or destroyed, these compounds act as emulsifiers and strong complexing agents for uranium and cannot be tolerated in the Purex system.

The organic acids can be degraded by oxidation to organic compounds that do not affect solvent extraction. Digestion of the raw feed with either 0.02 M $KMnO_4$ or 0.05 M $Ce(NH_4)_2(NO_3)_6$ at 70°C for 3 hr appears equally effective. After removal of the MnO_2 precipitate, the finished feed solution, when tested by exhaustive extraction with 30% tributyl phosphate in Adakane and exhaustive stripping with 0.01 M HNO_3, exhibited no unusual emulsification or interfacial crud. Uranium losses during extraction and stripping were negligible, being less than 0.01% in each step. Almost identical results were obtained when the feed was treated with ceric ion. The latter treatment may be preferred, since it does not require a feed-clarification step.

Infrared spectroscopy indicated that oxidation of the organic acids yields largely aliphatic compounds that may be partially nitrated. The path of these organic end products through solvent extraction has not yet been determined, pending the development of a suitable analytical procedure.

Fluoride Volatility Processing: Aluminum Alloy Flowsheet Development. – In continuing phase-equilibrium studies, liquidus isotherms in the system $LiF-KF-AlF_3$ at temperatures below 800°C were tentatively established. The 600°C isotherm includes $LiF-KF-AlF_3$ compositions containing more than and less than 25 mole % AlF_3. The minimum temperature in the quasi-binary system $3LiF \cdot AlF_3-3KF \cdot AlF_3$ was 645°C at 25.5 mole % $3LiF \cdot AlF_3$. In an attempt to obtain lower-melting solvents for AlF_3, alkaline-earth fluorides were added to $3LiF \cdot AlF_3-3KF \cdot AlF_3$ mixtures. Combinations of the alkaline-earth fluorides MgF_2, CaF_2, SrF_2, and BaF_2, whose optimal proportions have not yet been determined, lowered the solidus temperature for $3LiF \cdot AlF_3-3KF \cdot AlF_3$ mixtures by as much as 70°C.

Preliminary laboratory-scale tests designed to measure aluminum dissolution rates at 600°C in 50-50, 60-40, and 65-35 mole % LiF-KF were unsuccessful because of solids formation and subsequent foaming; solids formation began upon introduction of HF. The same effects were found in sparging 46.5-11.5-42.0 mole % LiF-NaF-KF (Flinak), thus tending to confirm the hypothesis that KF forms such a stable complex with HF that a phase transition is produced.

Fluoride Volatility Processing: Retention of TeF_6. – Gas-liquid absorption tests in packed columns 1 in. in diameter and 20 in. long were made in order to examine the behavior of TeF_6 in the Volatility Pilot Plant fluorine-disposal spray tower. During the four tests, about 10 mole % TeF_6 in fluorine flowing at 100 ml/min was passed through the columns countercurrent to 10% aqueous KOH. In two runs at ambient temperature, the absorption efficiencies appeared to be >20%; at a column temperature of 70°C, the efficiencies were >50%. However, these efficiency values are only approximate, since there was considerable uncertainty in the amount of TeF_6 introduced in each test. More-exact tests are planned.

Fluoride Volatility Processing: Dissolution and Fluorination in a Single Vessel. – Recent engineering-scale studies of the hydrofluorination and subsequent fluorination of Zr–6% U alloy fuel elements (simulated) in the same INOR-8 vessel showed a range of specific dissolution rates comparable to that found in previous studies, and no unusual fluorination or corrosion results. Six dissolution-fluorination sequences were made in 35-35-30 mole % $NaF-LiF-ZrF_4$ at 550°C with HF and F_2. Two additional fluorinations only were made after the completion of the six cycles. Specific dissolution rates varied between 0.8 and 1.55 mg cm^{-2} min^{-1}; fluorination half-times varied between 40 and 135 min. Some dissolution off-gas difficulties were experienced throughout the series, and the $HF-F_2$ inlet line failed during the startup of the fourth dissolution.

One INOR-8 and two alloy 79-4 corrosion specimens were suspended in the salt during five of the dissolution-fluorination cycles, the two additional fluorinations, and a cleaning step with aqueous ammonium oxalate solution. The 24-in.-long rods were suspended so that they penetrated the vapor-liquid interface. The maximum penetration by micrometer measurements for the INOR-8 was 11 mils in the interface region, while for alloy 79-4 it was 5 mils in the liquid region. The corrosion rods were in contact with molten salt for about 550 hr, during which HF was present for 64 hr and F_2 was present for 32 hr. Based on fluorine contact time, the corrosion rates for the rods were 0.33 and 0.16 mil/hr for INOR-8 and alloy 79-4 respectively. Vidigage measurements of the INOR-8 vessel showed

that maximum penetrations of 25 and 20 mils occurred in the vapor region and interface region respectively.

Fluoride Volatility Processing: Corrosion. – The dissolution of Zircaloy-2 with HF in 36.9-27.0-9.1-27.0 mole % $NaF-LiF-BeF_2-ZrF_4$ was found to be only mildly corrosive at 500°C when studied at Battelle Memorial Institute. Specimens of INOR-8, HyMu 80, "L" nickel, alloy 2 (10-5-85 wt % Mo-Fe-Ni), and Ni–5% Fe were tested. INOR-8, HyMu 80, and "L" nickel specimens with welds in which the respective base metals were used as a filler metal were tested, as were specimens of "L" nickel welded with INCO-41 and -61. The maximum corrosion rate by weight loss was 0.57 mil per month of HF exposure for "L" nickel welded with INCO-61. The rates for HyMu 80 and INOR-8 were similar, with a maximum of 0.27 mil per month of HF exposure. Significant intergranular attack was observed only on unwelded and self-welded "L" nickel and the Ni–5% Fe specimens. Difficulty was experienced in obtaining meaningful Zircaloy-2 dissolution rates over the entire run, but at 20 and 96 hr, rates were apparently only 0.4 and 0.2 mil/hr, respectively, based on H_2 evolution.

Memorandum BMI-X-218 (Feb. 15, 1963) presents the results of the examination of specimens removed from the Volatility Pilot Plant fluorinator after aqueous decontamination, and results of the simulated decontamination (ORNL-3410, p 9). The maximum base-metal corrosion rates for the specimens removed from the "L" nickel fluorinator were 0.38 mil per hour of fluorine exposure, including aqueous decontamination losses, and 0.26 mil per hour of fluorine exposure, excluding aqueous decontamination losses. Total molten-salt exposure for the fluorinator was 1920 hr; fluorine exposure was 57.5 hr.

BMI-X-218 also presents results of the metallographic examination of the corrosion specimens exposed in the Volatility Pilot Plant fluorinator during runs TU-11, TU-12, R-1 to R-6, and T-11 and the aqueous decontamination program. Salt exposure was 625 hr at 520°C maximum, including 20.8 hr of fluorine. The maximum total corrosion rates were 0.32, 1.83, 0.99, 1.31, 0.36, and 2.62 mils per hour of fluorine exposure for "L" nickel, Ni–1% Al, Ni–5% Mo, INOR-1, INOR-8, and HyMu 80 respectively. The high corrosion rate for HyMu 80 is attributed to attack by the $Al(NO_3)_3-HNO_3$ solution during the aqueous decontamination program.

Fluoride Volatility Pilot Plant. – After completion of fluorine leak testing of the Volatility Pilot Plant, inert gas was passed through the plant at temperature, using normal flow patterns. A salt-transfer test was then made. In spite of the aqueous decontamination program that had been carried out, radiation intensities at the waste-salt can ranged from 50 to 400 mr/hr.

Two complete runs with unirradiated fuel were made. In general, the runs were successful, but there were difficulties in melting the salt in transfer lines in both runs. Modifications should correct the difficulties. At least one more cold run will be required prior to hot operations.

In both runs the standard fluorination procedure (101 min at 6 std liters/min followed by 19 min at 13 std liters/min) was followed. In run TU-14 this procedure reduced the uranium content to an acceptable value (less than 10 ppm); however, in TU-13 only 99% of the uranium was volatilized, and an additional fluorination period was required. The reason for the incomplete fluorination during the first run is not known. In both

runs the movable absorber operated quite smoothly, and a 25% bed discharge at the end of each run added no uranium to the waste salt.

A molybdenum-stripping operation at 150°C was included in the UF_6 absorption procedure. In TU-13 the anticipated amount of molybdenum was removed, but the product was still contaminated with it. The source of this additional molybdenum has not been determined, and analyses from the TU-14 product are not yet available.

The product from both runs was contaminated with HF. An HF flashing operation was included in the product-handling procedure but apparently was inadequate. The source of the HF (several hundred grams in each run) has not been determined.

THORIUM UTILIZATION PROGRAM

Planning and Evaluation Studies. – Calculations (ORNL-3410, p 10) of the nuclear and economic potential of a 500-Mw (electrical) high-temperature graphite reactor were reviewed and extended. A large error in the estimation of the fission product losses was detected and corrected. For the cases considered, the minimum fuel-cycle cost was 0.82 mill/kwhr at a conversion ratio of 0.83. The maximum conversion ratio was 0.94 at a fuel-cycle cost of 1.21 mills/kwhr.

A study of unmoderated molten-salt reactors operating in the intermediate-to-fast neutron energy range was initiated. Such reactors have the advantage that they can employ halides of sodium and potassium instead of the more expensive Li^7 and beryllium salts. This permits salt discard as an economic means of minimizing the buildup of fission products.

Preliminary calculations were performed for a sodium-cooled core fueled with a molten mixture of plutonium and U^{238} chlorides (Cl^{37}), surrounded with an NaF-KF-ThF_4 blanket. The results indicate that a breeding ratio of 1.28 can be achieved at fuel-cycle costs of −0.3 mill/kwhr. The economic doubling time appears to be about 4.2 years.

A study of power generation costs in a 1038-Mw (electrical) molten-salt converter reactor as a function of the financing conditions was carried out. Conservative power-cost estimates in mills/kwhr for various types of ownership are: 2.09 (REA), 2.44 (municipal), 3.78 (investor owned), and 2.80 (Bodega Bay type financing). More-optimistic assumptions reduce these costs by 0.1 to 0.2 mill/kwhr.

MOLTEN-SALT REACTOR PROGRAM

Molten-Salt Reactor Experiment. – Fabrication of the MSRE heat exchanger and one fuel drain tank was completed in the Y-12 Shops. The second fuel drain tank and the radiator coil assembly are nearing completion.

Installation continued in the reactor cell and the drain-tank cell. The thermal shield and miscellaneous support structures were set in place. Considerable progress was made in the installation of auxiliary piping systems.

Specifications for the MSRE graphite were revised to permit minor defects in the finished graphite bars.

Metallurgy. – The MSRE heat-exchanger tube bundle was successfully welded, brazed, and inspected, using techniques previously developed for this program. The 326 weld joints were made semiautomatically without the addition of filler metal and inspected by radiographic and liquid-penetrant methods. No defects were found.

Brazing was accomplished in a dry hydrogen atmosphere having a flow of 345 ft^3/hr. A furnace temperature of 1850 to 1885°F was maintained for 1 hr. Both the heating rate and the cooling rate for the brazing cycle were about 300°F/hr.

A visual inspection of the completed tube bundle revealed good filleting and no apparent distortion. The metal was bright and clean. Ultrasonic inspection for unbonded areas was made by use of a Lamb-wave probe, with a 3/32-in.-diam flat-bottom hole as a standard.

Several tubes contained areas of scattered porosity in the braze that gave indications comparable to those from the reference hole. Duplicate conditions are being fabricated in samples in an attempt to further evaluate this condition metallographically. Despite the porosity, the brazed joints appear to provide an effective secondary leak seal for the heat exchanger and to strengthen the tube joint.

Chemical Development. – Revised and lower values for the solubility of oxide in MSRE fuel melts were associated with recently established solubility products: $[U^{4+}][O^{2-}]^2 = 1.2 \times 10^{-5}$ (moles/kg)^3 and $[Zr^{4+}][O^{2-}]^2 = 3.2 \times 10^{-5}$ (moles/kg)^3 in Li_2BeF_4 melts at 600°C. Saturation limits at as low as 80 ppm for precipitation of ZrO_2 from MSRE fuel were implied by these solubility products, which were obtained from changes in quadrivalent cation concentrations accompanying titration with added known increments of solid BeO.

A simple mass-action behavior consistent with the above solubility products was encountered, if data from attempts at a direct chemical analysis for oxide were not considered, in titrations of Li_2BeF_4 to which 1 or 5 wt % of MSRE fuel had been added initially. Such melts are representative of used flush salt, and even the small amounts of ZrF_4 that were present caused a marked lowering from the ~1100-ppm saturation limit for oxide ion in the unaltered Li_2BeF_4.

Preparation of MSRE Materials. – The operations by the Y-12 Plant to convert Li^7OH to Li^7F for use in preparing MSRE fluoride mixtures are progressing at a rate which should ensure completion by July 1963. Procedures for material transfer of some 12,000 lb of prepared Li^7F to the Reactor Chemistry Division were initiated. This material will be used in the coolant and flush-salt mixtures for the first reactor loading.

Some 240 kg of simulated MSRE fuel salt, LiF-BeF_2-ZrF_4-UF_4 (66.9-29.0-4.0-0.1 mole %), were produced. About 161 kg of this material will be used in the Engineering Test Loop studies; the balance is on hand for other experiments.

Studies of the reduction of FeF_2 from a molten LiF-BeF_2 mixture by hydrogen sparging indicate that the reaction rate is strongly dependent on the hydrogen flow rate and the temperature of the fluoride melt. The results of this study will be applied to the production processing techniques for removing structural-metal impurities from MSRE fluoride mixtures.

Radiation Effects. – The capsules containing MSRE fuel (25 g) and graphite which released F_2 at room temperature during shutdowns while under irradiation in the MTR have been monitored since the end of the in-pile exposure. The rate of production of F_2 decayed slowly, and indications were found of upper and lower temperature limits beyond which the release of F_2 virtually ceased, as shown below:

Cooling Period (days)	Temperature (°C)	cc of F_2 per day	
		0.7 mole % UF_4 fuel	0.35 mole % UF_4 fuel
12	36	22.7	13.5
15	36	13.5	7.0
20	36	10.0	5.1
27	50	7.6	5.0
35	86	0	1.6
42	34	5.1	3.3
46	–70	0.5	0
53	31	2.7	1.7

Seven of the 33 gas samples collected while the fissioning fuel was molten have still not been analyzed. Of two isolated instances of CF_4 among the analyzed samples of this type, a measurable rate of production of CF_4 was obtained during a 32-hr period, near the end of the sixth week of exposure, from the 0.7 mole % UF_4 fuel at a fission power density of ~70 w/cc and 810°C; this interval of ~32 kwhr produced 1×10^{-4} meq of CF_4 (6 ppm), which is low by a factor of more than 10 in comparison with rates that might be considered troublesome.

Analysis of Radioactive MSRE Fuels. – The coulometric method for the determination of uranium (a primary method) was tested on MSRE fuel with a 10-ml coulometric cell and the model Q-2005 coulometer; additional testing will proceed upon completion of instrumental checkout of the high-sensitivity coulometer, model Q-2564.

The apparatus for the pyrolytic determination of F^- (a primary method) was installed in the mockup; the determination was conducted on the MSRE fuel.

Several difficulties were encountered in additional testing of the method previously outlined for removing the powdered sample from the pulverizer-mixer. To overcome these difficulties, a new powder-transfer device was developed and fabricated. Manipulative testing of the new device and of the mixer mill in the moisture-free housing are being conducted in the mockup.

Fabrication of the transport handling equipment to remove the sample-filled copper ladle is approximately 90% complete. Other phases of the work necessary for handling and analyzing the MSRE fuel sample in the hot cell are in progress.

The evaluation of the coulometric method for the determination of uranium in sulfuric acid solutions of MSRE salt was completed. With simulated hot-cell operation, a relative standard deviation of 0.5% was obtained for determining uranium in unirradiated MSRE salt solutions.

The evaluation of the pyrolytic determination of fluoride in MSRE salt was completed; the evaluation was carried out in the Analytical Chemistry mockup cell, using an apparatus designed for remote operation; a relative standard deviation of 1% was obtained for unirradiated MSRE salt.

ADVANCED REACTOR DEVELOPMENT

Advanced Test Reactor Corrosion Program

Most of the tests designed to determine the effect of heat flux on the formation of corrosion-product films on aluminum alloys have been carried out with the pH of the cooling water adjusted to 5.0 with nitric acid. In addition, the heat flux across the aluminum-water interface has usually been between 1×10^6 and 2×10^6 Btu hr^{-1} ft^{-2}, and the coolant velocity has ranged from 35 to 45 fps. Under these conditions, the thickness of the corrosion-product layer formed on the aluminum surface was related to exposure time and temperature at the specimen-water interface by an equation of the form,

$$X = a\theta^b \exp(-c/K) ,$$

where X is corrosion-product thickness, θ is exposure time, K is temperature, and a, b, and c are constants. The best values of the constants, estimated from a linear regression analysis of the experimental data, are a = 443, b = 0.78, and c = 8290, with X in mils, θ in hours, and K in degrees Kelvin.

The average value of the thermal conductivity of the corrosion-product oxide derived from 79 separate determinations was 1.3 Btu hr^{-1} ft^{-2} $(°F)^{-1}$ ft, with a standard deviation of 0.2.

High Flux Isotope Reactor Design and Development

Fuel Element Development. — An inner annulus using dummy aluminum plates was assembled and met both the average-plate-spacing and maximum-spacing-deviation specifications. The prime purpose of the test was to demonstrate that the fuel plates could be attached to the outside of the inner tube by circumferential welds made in grooves cut inside the tube. Grooves were cut circumferentially at 1-in. intervals down the length of the tube. In spite of the limited space, all welds were made successfully, using a specially developed automatic welding head.

A technique has been developed by which both the gradient fuel core and the insert containing the burnable poison may be fabricated as an integral piece in a single pressing operation, producing more homogeneous and cheaper fuel plates. Plates using such cores have been fabricated and met all tolerances. This procedure will be tested on a pilot-plant basis in fabricating the fuel plates for the next inner annulus.

Design and Analysis. — Final detail drawings of the beryllium reflector were certified for construction, to permit final machining of the rough-machined pressings on hand.

In order to reduce friction in the control region, the outer control-rod guide bearings were changed from skids to ball bearings. The guide tracks are being redesigned to accommodate the ball bearings. A test program is under way to evaluate the new bearings. Hydraulic experiments are being conducted to determine the bearing loads.

Tests of the prototype pneumatic ball-latch actuator in the outer control-rod drive showed that the system response time under typical operating conditions is approximately 11 msec with three circuits scramming and 26 msec with two out of three circuits scramming. The design criteria for the system response time is 10 msec. It is estimated that the response time could be reduced to approximately 12 to 15 msec by the addition of another component and by considerable additional development; however, in view of these initial results, it was decided that a magnetic actuator would more likely provide the desired response time with greater reliability. Design of the magnet actuator is under way and includes a spring-loaded snubber which allows for thermal expansion or contraction of the push rod.

Engineering Development. — Flow tests have been conducted on the HFIR mockup at flow rates ranging from 2100 to 17,000 gpm. More instrumentation was provided to obtain additional pressure data, particularly in the control-region area and the vessel outlet region. The only strain gages monitored were those affixed to the N^{16} and fission-chamber probes. Flow tests are continuing, utilizing a new target assembly.

Three bearing testers have been fabricated for testing bearings and bearings materials specified for use on the HFIR control-rod drive extensions and on the HFIR inner and outer control rods. The testers are equipped to duplicate reactor coolant-water temperature and pressure conditions. Tests will provide information on bearing wear, friction, and life as a function of load and exposure time.

A seal-test device is being operated to evaluate various types of seals which may be suitable for sealing against leakage of reactor water around the 0.375-in.-diam ball-latch-mechanism push rod. The results of this test will provide a basis for establishing preventive maintenance procedures based on the life expectancy of the seal.

Instrumentation. — A prototype of an on-the-line analog computer has been built and tested to solve for the loss of reactivity due to xenon growth and fuel depletion. Tests of the prototype indicate that the overall accuracy obtainable, including long-term drifts, is approximately 3% of the total reactivity contribution due to xenon and fuel depletion.

The feasibility having been thus established, further development incorporating other features, such as the effect of samarium, will lead to the production of a computer to be incorporated in the HFIR control system to determine when the shims may be safely withdrawn at the request of the servo control system.

PHYSICAL RESEARCH PROGRAM

Reactor Operations. – During the first nine weeks of 1963 the ORR operated 70.7% of the time. Loop No. 2, a gas loop for testing unclad or partly clad fuel elements, required a specially extended shutdown of almost three weeks. A dummy fuel element was received from the new ORR fuel fabricator, Sylcor Division of Sylvania Corporation, and was accepted. Two fission channels for startup are now installed at the ORR.

At the LITR, about one-third of the instrument modification has been completed.

Approximately one-third of the irradiated ORNL Graphite Reactor fuel stored in the canal has been shipped to Savannah River for reprocessing.

A set of operating limits for the ORR was prepared and was discussed with the AEC; final revision is being made.

Waste Disposal. – The release of Sr^{90} from the process-waste system into White Oak Creek remained steady for the last six months at 0.2 curie/month. During the same period, the releases from other, unknown sources have averaged 0.4 curie/month. A continuing investigation of the many creek discharges has failed to reveal the main source of the activity.

Hot-Cell Operations. – Operations in the hot cells were normal. Two meltdown experiments were disassembled – one containing UC_2 particles embedded in graphite and the other containing UO_2. Several GCR fuel capsules from the ORR and the LITR were examined; the fuels included UC_2 particles, in graphite, and UO_2. One MSR capsule containing UO_2 and one thorium-utilization capsule containing UO_2-ThO_2 were examined. Several tube-burst specimens, APPR miniature absorber (Eu_2O_3) plates, thoria sol-gel pellets, and HRT core-tank pieces were also examined. Specimens were recovered from 18 ORR experiments in the ORR cells. Capsules from molten-salt experiment MTR-47-4 were sectioned and examined; experiment MTR-47-5 was received and pressure measurements for gas buildup at various temperatures were begun. Disassembly and examination of assembly ORNL 41-8, containing encapsulated BeO pellets, were begun.

PHYSICS AND MATHEMATICS RESEARCH

Charge Spectrometry. – The relative abundances of fragment ions formed following the β^+ decay of CH_3F^{18} have been measured with a specially designed mass spectrometer. The following ions with their percent abundances were observed: CH_3O^- (12), O^- (8), H^- (17), CH_3^+ (39), CH_2^+ (13), CH^+ (5), C^+ (2), and H^+ (4). Also, the production of a rather large number of neutral species is suggested by an apparently low total yield of ions. The large percentage of positive ions seems inconsistent with the predicted amount of electron shake-off following positron decay, at least for atomic systems. There is a possibility, however, that when the collection chamber is at a positive potential, CH_3O^- might strike the walls of the chamber with sufficient force to create CH_3^+.

Neutron Diffraction. – Neutron diffraction studies on the alloy system Dy-Y have been made to investigate the effect of dilution on the magnetic structure properties of dysprosium. The addition of yttrium stabilized the helical antiferromagnetic phase of dysprosium at the expense of the ferromagnetic structure. As little as 5% yttrium keeps the alloy from becoming completely ferromagnetic at 4°K, although there does appear to be a ferromagnetic component present in the 95% Dy–5% Y alloy. The Néel temperatures of the alloys are quite closely proportional to the 2/3 power of the atomic concentration of dysprosium. The interlayer turn angle of the helix increases with increasing yttrium content and approaches a limiting value near 50° per layer at low temperatures. In alloys with the yttrium concentration greater than about 50 at. %, the interlayer turn angle is very nearly independent of temperature.

Theoretical Physics: Pickup of Heavy Clusters. – It has been suggested that the existence of alpha-particle clusters in nuclei might be determined by a study of the (d,Li^6) reaction, or its inverse. Accordingly, Denes and Daehnick measured angular distributions of Li^6 ions produced by deuteron bombardment of C^{12}, O^{16}, O^{18}, and F^{19}. These experiments also revealed formation of Li^7 ions with cross sections comparable to those for the production of Li^6. The angular distributions show forward peaking, suggesting a pickup process. These data were analyzed in distorted-wave approximation; an extreme cluster model was assumed for the heavy cluster and the residual nucleus. Reasonable agreement between experimental and theoretical angular distributions was found.

Theoretical Physics: Elastic and Inelastic Scattering of Carbon from Carbon. – The measurements of Wang *et al.* and Garvey *et al.* on the scattering of 127-Mev carbon ions from carbon show, in addition to elastic scattering, strong single excitation of the 4.43-Mev (2^+) state in one of the nuclei, the single excitation of the 14-Mev (4^+) state, and the "mutual" excitation of both nuclei to the first excited state. Assuming the two-body effects to be predominant, the data were analyzed for the single excitations in terms of the collective model: (1) in distorted-wave approximation and (2) in strong-coupling approximation, where the rotational model is assumed. Good agreement was found between theory and experiment, both in absolute magnitude and in the shape of the angular distributions.

Fast-Chopper Time-of-Flight Spectrometer. – The gamma rays from thermal and resonance neutron capture were studied with a large (9-in.-diam × 12-in.-long) sodium iodide crystal, with its associated shielding, and the fast-chopper spectrometer at the ORR. A flight path of 5 m results in a neutron energy resolution of about 15% up to 200 ev. Gamma-ray spectra were measured from one or more resonances as well as from thermal-energy neutrons in each of the seven abundant tin isotopes. In general the gamma-ray spectra from the resonances are much simpler than the thermal spectra. For example, the gamma spectrum from the 62-ev resonance in Sn^{124} consists almost entirely of a strong gamma ray at 5.7 Mev and a weak one at ~4.7 Mev. This strong gamma ray probably consists of transitions in Sn^{125} to the $1/2^+$ state at 0.19 Mev excitation and the $3/2^+$ excited state at ~0.03 Mev. Similarly, in Sn^{118} the gamma-ray

spectrum from the 45.8-ev resonance shows a strong gamma ray at 6.6 Mev, probably consisting of transitions to the $1/2^+$ ground state and the $3/2^+$ excited state at 0.024 Mev. The gamma spectra change appreciably from resonance to resonance in a single nuclide.

Low-Temperature, Nuclear and Solid State Physics. – In the Mössbauer effect, there is a shift of the gamma-ray energy with the s-state charge density on the atom. This s-state charge density may be modified if the atom is alloyed with other metals. A study has been made of alloys of gold with nickel, palladium, platinum, silver, and copper in which the Mössbauer isomer shift of the gold and also the residual electrical resistance associated with the gold have been measured. It is found that the above isomer shift and electrical resistance may be correlated through a quantum mechanical model. The method offers a new approach to the study of the s-band structure in dilute alloys.

High-Voltage Experimental Program: Elastic Scattering of Alpha Particles by O^{18}. – The differential cross section for elastic scattering of alpha particles by O^{18} has been measured for alpha-particle energies between 2.4 and 3.4 Mev and for $\theta_{c.m.}$ = 90, 125.3, 140.7, and 152.3° in order to determine the level parameters of excited states in Ne^{22}. Thin targets (< 2 kev for 2.4-Mev alphas) of O^{18} were prepared by oxidizing tungsten in an H_2O^{18} atmosphere and then evaporating the oxide onto thin carbon backings. The scattered alpha-particle energy was measured with a double-focusing, uniform-field reaction-product magnet. Previous work [Phys. Rev. 128, 299 (1962)] on the $O^{18}(\alpha,n)Ne^{21}$ reaction indicates the presence of eight resonances in the above energy range corresponding to excitation energies in Ne^{22} between 11.686 and 12.38 Mev. Six of these eight resonances have been observed in the scattering data; preliminary results give $J^\pi = 2^+, 1^-, 1^-, 0^+, 0^+$, and 2^+, corresponding to the resonances at E_α^{lab} = 2.47, 2.57, 2.72, 2.90, 3.19, and 3.32 Mev respectively. Qualitative fits to the data, using dispersion theory, have been obtained for three of the six levels.

High-Voltage Experimental Program: Heavy-Ion Energy Accumulation in the Tandem Van de Graaff Accelerator. – A new energy accumulation method was found for accelerating very small but usable beams of artificial fission fragments such as bromine and iodine ions to energies three times higher than those previously available from tandem Van de Graaff accelerators. Energies up to 120 Mev now available cover completely the energy range found for the fission particles from uranium. The ions have been used to study the behavior of silicon solid-state fission-particle detectors of many types. For the first time, direct calibration methods are available to aid in experimental studies of the fission process. Further work will be directed toward an experimental study of the stopping power of various elements for heavy ions.

CHEMISTRY RESEARCH

Effects of Radiation on Analytical Methods. — The study on the effect of gamma radiation on electrode systems has been continued. This work has been hindered by the apparent instability of the salt bridge under the conditions necessary to carry out these studies inside the gamma source.

An investigation on the effect of gamma radiation on the dimethylglyoxime method for determining nickel colorimetrically was initiated. Nickel solutions (0.001 M) have been exposed to ~10^5 r of gamma radiation with no apparent change in the nickel concentration.

Chemical Engineering Research. — A new design series of stacked-clone contactor, designated the Mark X, was built in its first conformations and tested with the system 1 M $NaNO_3$, $UO_2(NO_3)_2$, 18% tributyl phosphate in Amsco. The contactor exhibited flooding throughputs of > 4.5 liters/min at stage efficiencies of 60 to 65%, giving a "performance factor" (throughput × efficiency) of 2.7 to 2.9. This compares with a factor of 2.3 for the best performance of previous contactors (Mark VI). The new series has features suggested by previous tests: an underflow skirt, a mixing chamber between the underflow port of one stage and the vortex finder of the stage below, and a baffled passage between the mixing chamber and the pump suction plenum designed to promote radially symmetrical flow.

Solvent Extraction Research. — In comparative tests with several solvents, Am(III) was usually extracted preferentially to Am(VI) from nitrate solutions. Single-stage separation factors attained from various systems included: > 10,000 with di(2-ethylhexyl)phosphoric acid from nitric acid, 100 with tri-n-octylphosphine oxide from sodium nitrate, and 30 with tributyl phosphate (TBP) from lithium nitrate. In extractions by tertiary amines, the differences were smaller and sometimes were in the opposite direction. These data indicate possible applications to separation of americium from curium, which cannot be oxidized in solution and which usually extracts slightly better than Am(III).

Continued studies of strontium extraction from sodium nitrate solutions by di(2-ethylhexyl)phosphoric acid ($[HA]_2$) and its sodium salt (NaA) in benzene confirmed the previously suggested reaction $Sr^{2+} + 3[HA]_2 \rightleftharpoons SrA_2 \cdot 4HA + 2H^+$ (ORNL-3331), and also the corresponding reaction $Na^+ + 2[Ha]_2 \rightleftharpoons NaA \cdot 3HA + H^+$, when the extractant is preponderantly in the acid form, $[HA]_2$. When the extractant contains significant amounts of the sodium salt, the generalized equation, $Sr^{2+} + (n/y)\ [aHA \cdot bNaA]_y \rightleftharpoons SrA_2 \cdot (n-2)(\alpha HA \cdot \beta NaA) + iH^+ + jNa^+$, $a + b = 1$, $\alpha + \beta = 1$, $i + j = 2$, has proved to be a suitable model at least up to 50% salt form. Experimental evaluation of i from H^+ concentration dependence, n/y from extractant concentration dependence, and y from vapor-pressure measurements (ORNL-3368) leads to calculated values of n in the range 4 to 6, indicating that the extracted strontium is in the form of $SrA_2 \cdot 4HA$ or analogous adducts throughout this region.

Triphenylmethane was used as a reference standard in relative osmotic measurements of organic solutions by the isopiestic-vapor-balancing

technique (ORNL-2166, -2578), although there was evidence of some deviation from ideality (ORNL-2845). Its activity coefficients in benzene solution have now been determined by the direct differential-vapor-pressure technique (ORNL-3120), varying from 0.98 at 0.014 m to 0.74 at 0.33 m. Activity coefficients of azobenzene, another useful reference solute, were then determined in benzene solution by isopiestic balancing with the triphenylmethane, ranging from 0.96 at 0.0134 m to 0.67 at 0.346 m. Thus, both of these reference solutes are now available for absolute as well as relative isopiestic measurements.

The efficiency of a pulsed solvent extraction column at a high aqueous-to-solvent flow ratio (100:1) was tested with 30% TBP to extract uranyl nitrate from 4 M $NaNO_3$ solution. The height equivalent to a theoretical stage of a nozzle-plate cartridge (0.125-in. holes, 10% free area), operated with the organic phase continuous, was 6.3 ft at a pulse of 35 cpm and a 1-in. amplitude.

Process Chemistry of Uranium and Thorium. – Beryllium was recovered from ore sulfate leach liquors (pH about 2.5) by extracting with primary amines. Of the compounds tested, best results were obtained with 1-(3-ethylpentyl)-4-ethyloctylamine. The beryllium extraction coefficients increased with increase in sulfate concentration in the range 0.5 to 2 M and were higher for 10-min than for 60-min contact times.

Results of preliminary tests show possibilities for applying solvent extraction with amine reagents to the recovery and purification of phosphoric acid from commercial phosphoric acid. Tertiary amines with a moderate degree of steric hindrance, for example, benzyldilaurylamine and 2-ethylhexyldilaurylamine, showed the best balance between phosphoric acid extraction power and ease of stripping with water.

Volatility Studies. – A method for preparing granular LiF with a high specific surface area was developed for use in PuF_6 studies. It consists in fusing Li_2CO_3, solidifying, and then slowly fluorinating with elemental F_2 at ambient temperature. A surface area of about 40 m^2/g was obtained with the material, and it had sufficient granular strength for use in adsorption beds. Granular LiF with an area of only 1.6 m^2/g was obtained from ORGDP for comparative tests in the PuF_6 work. It was prepared from analytical-grade powder by a wet-compaction and drying method. The fusion-fluorination method may be useful in the preparation of other sorbent fluorides of high surface area.

The first test with a stirred reactor on the volatilization of PuF_6 from fused LiF-NaF-ZrF_4 was unfavorable, with the volatilization rate being approximately the same as in previous static tests. However, this test lasted less than an hour, and it was evident that further improvements in the design of both the stirrer and the reactor were desirable.

Ion Exchange Studies. – Citric acid was evaluated as a complexing agent for Al^{3+}, Fe^{3+}, and Cr^{3+} at a pH of 2.5 – a higher acidity than is routinely considered. Bulk quantities of these cations interfered with the direct use of cation exchange for recovering useful fission products from radioactive waste solutions. For the specific case of Purex IWW waste, the capacity for strontium was less than one resin volume of solution, because the molar concentration of iron in the waste was more than

100 times that of the strontium. Previous studies with oxalic acid indicated that it complexed iron effectively and, correspondingly, increased the resin capacity for strontium threefold. However, oxalates precipitated on the resin bed as the acidity of the local exchange zone was lowered to pH's between 0 and 2 by the displacement of hydrogen ion with such sorbable cations as the rare earths and strontium.

Distribution coefficients were determined for rare earths (Ce^{3+}), Sr^{2+}, and Fe^{3+} in the acid concentration range 0.1 to 1.1 N, corresponding to Purex IWW waste dilutions of 5 to 40. Major cationic impurities were complexed by (1) adding citric acid in the ratio of 1.5 moles per mole of combined Fe^{3+}, Al^{3+}, and Cr^{3+}, (2) adjusting to pH 2.5 with concentrated ammonia, and (3) heating the solution to 90 to 100°C for 0.5 hr to ensure complete complexing of the chromium ion. The resin used in the experiments, Dowex 50W-X8 (100 to 200 mesh), was equilibrated with a 0.5 M HNO_3–0.005 M ammonium citrate solution at pH 2.5 prior to its use.

Distribution coefficients for both strontium and rare earths increased as the acidity was lowered by dilution. Strontium values followed a second-power dependence, as predicted for a divalent cation, ranging from 10 to 800 as the acidity was lowered from 1.1 to 0.1 N. Rare-earth (Ce^{3+}) distribution coefficients varied from 3 to 2300 over the same acid concentration range, but did not follow the predicted third-power dependence for a trivalent cation. Ferric ion was very strongly complexed by the citrate ion even at a waste dilution factor of 400 (0.014 N), while the distribution coefficient remained less than 0.3. Plotted experimental data indicated that at a reasonable dilution factor (20), more than three volumes of original Purex IWW could be processed per volume of resin before strontium breakthrough and that favorable separation of the various fission products from each other and from iron would occur, that is, K_d^{Sr}/K_d^{Fe} greater than 200, K_d^{Ce}/K_d^{Fe} greater than 600, and K_d^{Ce}/K_d^{Sr} greater than 3.

Mechanisms of Separations Processes. – In a continuation of studies of the thermodynamics of extraction of uranyl nitrate and nitric acid by tributyl phosphate, additional measurements of the partial pressures of water and acid over the system H_2O-HNO_3-$UO_2(NO_3)_2$ were made. Acid concentrations ranged from 3 to 17 m, and uranyl nitrate from 0 to 1.8 m (near saturation).

To calculate the activities of uranyl nitrate from the measured activities of water and nitric acid, the Gibbs-Duhem equation must be integrated for this three-component system. It was found here that by appropriate choice of the paths of the integration, the integration can be performed exactly and that the final equation assumes a very simple form:

$$(n_2/n_1)\ \ln\ [a_2(n_2,n_3)/a_2(n_2,0)] + (n_3/n_1)\ \ln\ [a_3(n_2,n_3)/a_3(0,n_3)]$$
$$= \ln\ \{[a_1(n_2,n_3)]/[a_1(n_2,0)a_1(0,n_3)]\}\ , \qquad (1)$$

where subscripts 1, 2, and 3 refer to H_2O, HNO_3, and $UO_2(NO_3)_2$ respectively; n_i is the number of moles of component i in the solution; $a_i(n_2,n_3)$ is the activity of component i in a solution containing n_2 moles of HNO_3 and n_3 moles of $UO_2(NO_3)_2$. A zero within parentheses indicates that a component is not in the solution; for example, $a_1(0,n_3)$ is the activity of water over H_2O-$UO_2(NO_3)_2$ solution.

Measurements of water-vapor pressure over 26 solutions show that $[a_1(n_2,n_3)]/[a_1(n_2,0)a_1(0,n_3)]$ is 1.008, with a standard deviation of 0.034; that is, this term is unity, within experimental error. Thus, the term on the right of Eq. (1) is zero, and the activities of uranyl nitrate can be calculated in the three-component solutions from the measured activities $[a_2(n_2,n_3)]$ of nitric acid in the three-component solution and literature data on activities of nitric acid in the two-component solution H_2O-HNO_3 [i.e., $a_2(n_2,0)$] and activities of uranyl nitrate in the two-component solution H_2O-$UO_2(NO_3)_2$ [i.e., $a_3(0,n_3)$].

Experimental data show that the term $n_2 \ln [a_2(n_2,n_3)/a_2(n_2,0)] = - n_3 \ln [a_3(n_2,n_3)/a_3(0,n_3)]$ is nearly (but not exactly) a linear function of m_3, the molality of $UO_2(NO_3)_2$ in the solution. They show that, to a first approximation, nitric acid does not change the activity of uranyl nitrate (at constant uranyl nitrate molality), although uranyl nitrate increases the activity of nitric acid. These results may be summarized as follows:

$$\ln [a_2(n_2,n_3)/a_2(n_2,0)] \cong 5(m_3)^x/m_2 \ , \qquad (2)$$

where x may be nearly (but not identically) unity.

Although further analysis of the data is necessary, the form of Eq. (2) shows that the three-component system H_2O-HNO_3-$UO_2(NO_3)_2$ does not follow the Harned rule for interaction of two solutes in water.

The integration of the Gibbs-Duhem equation was extended to aqueous solutions containing three solutes, and the procedure can readily be extended to even more complex solutions. It therefore appears probable that the present thermodynamic study can be easily extended to extraction of trace components (such as fission products, and perhaps studied one at a time) from H_2O-HNO_3-$UO_2(NO_3)_2$ solutions.

Physical Chemistry of Ion Exchangers. – Isopiestic-vapor-pressure measurements were completed on tetramethyl ammonium, tetraethyl ammonium, tetrapropyl ammonium, and tetrabutyl ammonium chlorides and bromides. Tetramethyl, tetraethyl, and tetrapropyl ammonium iodides were also measured. In dilute solution (below 0.5 m) the osmotic coefficients for the chlorides decreased with the size of the cation: $Bu_4N^+ > Pr_4N^+ > Et_4N^+ > Me_4N^+$; for the bromides the reverse order is found: $Me_4N^+ > Et_4N^+ > Pr_4N^+ > Bu_4N^+$. Similarly, for the iodides the order observed was $Me_4N^+ > Et_4N^+ > Pr_4N^+$.

At higher concentrations the osmotic coefficients of the larger cation salts decrease sharply, and some of the orders given above are reversed. This behavior suggests that aggregation or micelle formation occurs.

Nuclear Chemistry: Fission Studies and Characterization of Low-Energy Properties of Nuclei. – The energy spectrum of the neutrons following beta decay of the 4.1-sec delayed-neutron emitter N^{17} was studied by the time-of-flight technique. A beta-ray event was detected in a plastic scintillator and announced the emission of a neutron. The neutron traversed a flight path of either 50 or 100 cm and was detected by its proton recoil in another plastic scintillator. The interval between the beta and neutron events was converted to pulse-height information by

using a modified Neiler-type time-to-height converter. The N^{17} was produced by reactor irradiation of the compound $Li_3^6N^{15}$, which caused the reaction $N^{15}(t,p)N^{17}$ to take place. Three neutron groups were observed with center-of-mass energies of 404 ± 10, 1213 ± 20, and 1807 ± 40 kev. The results indicate neutron emission from the 5.94 ($1/2^-$), 5.38 ($3/2^-$), and 4.56 ($3/2^-$) Mev levels of O^{17}.

Organic Chemistry. – The gas chromatographic separation of the butene isomers formed in the pyrolysis of butyl phosphates was investigated by using a number of high-boiling liquids (individually) on Celite at 25°C. The objective was to find a substitute for the relatively volatile dimethyl formamide, which must be used at 0°C. Several amides, amines, ethers, nitro, organophosphorus, and other compounds successfully resolved the butene mixture, but the best solvents were found to be o-nitrophenetole ($NO_2C_6H_4OC_2H_5$) and triethyl phosphate [$(C_2H_5O)_3PO$]. A 13-ft column of o-nitrophenetole on Celite, operated at 25°C, gave separation factors of 1.00, 1.35, 1.57, and 1.98 for butene-1, trans-butene-2, cis-butene-2, and 1,3-butadiene, respectively, where butene-1 emerged 4.5 min after the air peak.

Electrochemical Kinetics and Its Application to Corrosion. – An investigation of the electrochemical behavior of iron in hydrogen-saturated solutions of sulfuric acid and sodium sulfate over the pH range 0.3 to 5.0 was completed. Equations relating the rates of the iron dissolution and hydrogen evolution reactions to pH and electrode potential were obtained. Expressions which directly relate corrosion potential and corrosion rate to pH were also obtained. Characterization of the behavior of iron in the noninhibitive sulfate solutions was a necessary prerequisite to the understanding of inhibitor action.

By comparing the results obtained in the noninhibitive sulfate solutions with those obtained in inhibitive benzoate solutions, it was shown that in the latter the benzoate anion is strongly adsorbed on the iron. The adsorption of the anion results in an acceleration of the hydrogen evolution reaction and a retardation of the iron dissolution reaction. The anion adsorption is potential-dependent. Consequently, the anion can be removed from the iron surface by sufficiently large cathodic or anodic polarization. The behavior of iron in the benzoate solutions then approximates that found in the sulfate solutions.

Physical Chemistry of Molten Salts. – Activity coefficients of ZrF_4 in the system $LiF-ZrF_4$ were evaluated in order to obtain information on the thermodynamic solution behavior of ZrF_4 in a fluoride melt not too different in character from MSRE fuel. From 1.0 to 0.2 mole fraction of ZrF_4, activity coefficients were calculated from published vapor-pressure information. From 0.2 to 0 mole fraction, the activity coefficients were calculated by means of the Gibbs-Duhem equation. To apply this equation, the necessary activity coefficients of LiF were determined for this concentration range from measurements of liquidus temperatures. The activity coefficients of ZrF_4 for the concentration range of reactor interest (5 mole % and less) were calculated to be of the order of 10^{-5}.

Chemical Separation of Isotopes. – The solubility of BF_3 in nitrobenzene, tetrahydrofuran (THF), triethyl amine, and thiophenol was determined. At 760 mm and room temperature, thiophenol dissolves only trace amounts of BF_3, while Et_3N dissolves only enough to form the 1:1 addition compound. Values for nitrobenzene and THF are shown below. Results are in moles of BF_3 dissolved per mole of organic:

760 mm Hg				400 mm Hg			
Nitrobenzene		THF		Nitrobenzene		THF	
25°C	0.0465	22°C	1.0316	25°C	0.0253	22°C	1.005
20°C	0.0600	14°C	1.0476	20°C	0.0335	14°C	1.0111
15°C	0.077	8°C	1.0622	15°C	0.0438	8°C	1.0206
5°C	0.1250			5°C	0.0746		

The single-stage isotopic equilibrium constant was determined for the reaction

$$B^{10}F_3(gas) + B^{11}F_3\cdot\langle\bigcirc\rangle\text{–}NO_2(liq) \rightarrow B^{10}F_3\cdot\langle\bigcirc\rangle\text{–}NO_2(liq)\ .$$

This value is 1.032 at 7°C, with B^{10} concentrating in the liquid phase.

Oxygen-17 Pilot Plant. – The O^{17} separation facility continues to perform well. The isotopic gradient in the water distillation cascade is developing as expected. After 34 weeks of operation, the O^{17} and O^{18} concentrations have reached 1 and 40% respectively.

Chemical Applications of Nuclear Explosions. – Water-leaching studies of a large composite sample of known particle-size distribution from the Gnome event showed that the water-soluble material is dissolved in 30 min or less. The percentages of water-insoluble residue remaining after leaching at room temperature were 8.8, 8.1, 7.1, 6.0, and 5.8 for water-to-sample-weight ratios of 2.8, 3.3, 4.2, 5.6, and 6.9 respectively. The quantity of insolubles was about 20% less in 30-min leach tests than in 24-hr leach tests for water-to-sample ratios less than 5.3, indicating that it is possible to supersaturate the solutions in short leach periods. Removal of activity by nitric acid leaching is independent of acid concentration above 1 N when excess acid is used. Acid consumption was 15 to 20 meq per gram of residue leached. Although ferric hydroxide was shown to be an effective scavenger of Am, Cm, Pu, and Eu from leach solutions, at pH's above 6, the leach solutions contained too much ferric iron to give good decontamination factors. When all of the iron was precipitated, the ferric hydroxide retained about a third of the solution volume in its gel structure. Oxalate precipitation of Ce^{3+} carrier or of very small accounts of calcium quantitatively carried $Eu^{152-154}$ tracer from 1 N HNO_3 solutions saturated with $CaSO_4$ in which the ferric ion concentration was varied from 0.5 to 1.5 mg/ml. The pH of the solutions was

adjusted to 1.0 to 1.5 with NaOH to cause precipitation. Cerous ion concentrations as low as 100 ppm, or calcium in this equivalent, were used to quantitatively carry the europium as oxalate.

METALLURGY AND MATERIALS RESEARCH

Physical Properties Studies. — A thermal-conductivity determination on 93.4%-dense UO_2 in the improved radial-heat-flow apparatus has progressed through the following stages: (1) an initial heating to 1300°C, (2) a cooling to 1050°C, (3) a reheating to 1150°C, (4) a cooling to 900°C, and (5) a reheating to 1225°C. This cycling represents a significant improvement in the performance of the apparatus, since previous determinations were terminated by component failures at high temperatures on the initial heatings. The results to 1050°C exhibit a spread of ±0.5%, and the thermal resistance (1/K) is linear to ±1%, as expected for pure phonon conduction. Above 1050°C the data scatter increased to ±2%; however, on a 1/K plot the results are definitely below the extrapolation on the low-temperature line. This deviation is as much as 6% at 1300°C and is thought to be due in part to supplementary heat transfer mechanisms such as infrared transmission or bipolar diffusion. The 1/K results obtained at 900 and 1050°C on cooling from 1300°C were 1 to 2% below the values obtained on heating. This shift indicates that an irreversible change of the specimen, such as in its stoichiometry, occurred at the high temperatures. This phenomenon is being studied during the reheating now in progress.

CONTROLLED THERMONUCLEAR RESEARCH

DCX-1 Facility. — In order to determine the stabilizing influence of end plates on the DCX-1 spreading mechanism, movable end walls capable of being biased up to ±15 kv were installed. Subsequent experiments showed that within the range −15 to +15 kv, the plasma was in no way stabilized by either the position or the potential of the plates. At the high voltage both plasma density and containment time were decreased by a factor of 2. Optimum plasma density is obtained by having the end plates at ground potential and removing them to a position at least 6 in. from the median plane.

Additional measurements of the loss of fast protons from each mirror of an asymmetric mirror were made. A 20% short in the east mirror coil caused the asymmetry, and measurement revealed that twice as many protons escaped through the "leaky" mirror coil as through the strong coil; however, this loss rate is still down a factor of ~100 from the calculated rate. Correlations between the rf bursts at low pressures and the particles lost through the mirror could not be seen.

In order to separate the effects of the concentric proton motion (ordered) and of precessive proton motion caused by the radial field gradient, an obstacle was positioned radially so that it intercepted the

desired region of plasma. The results show that half of the measured plasma density results from protons that precess through the central 1-in. region of the machine, that the precessive portion of trapped protons contributes in no discernible way to the generation of ion clumps, and that the measured energy dispersion arises mainly from the ordered proton motion. This experiment substantiates other experiments which have shown that substantial quenching of the plasma spread can be achieved by introducing more precession by raising the magnetic field for fixed injection angles.

Detailed correlation studies of the initial portion of the accumulation and decay of the plasma with the rf bursts show that in addition to the time delay for the plasma reaching the large Z positions, there exists a time delay of 0.2 sec before the plasma at large Z begins to decay. The inference is that as long as the instability persists as indicated by the presence of ion clumps, there is a flow of particles to large Z; once the ions cease clumping, the loss everywhere in the plasma proceeds by charge-exchange loss rate.

Modifications and repairs, which have consumed two weeks, included the replacement of the 20%-shorted east mirror coil, the sealing of a leak in the liner region that prevented attaining pressure below 3×10^{-9} torr, the installation of a magnetic quadrupole lens to focus larger injected currents, and changes in the ion source that will permit the extraction of higher currents.

High-Current Injection. — Beam accountability studies have been conducted with the DCX-2 injector system. Calorimetric measurements were made of the power emerging from the injector and the power dissipated inside under various operating conditions. The beam accountability was good under conditions where a large fraction of the beam was passed through the injection duct and was poor when a large fraction was dissipated inside, for reasons now understood. These studies were limited to a maximum total current of only 75 ma, and the maximum H_2^+ fraction injected into the machine was 37 ma. The reason for this discrepancy as compared with previous laboratory analysis from this same ion source, where 65 to 70% of the beam was H_2^+ at this current level, has not been determined. A new lens coil has been obtained for the high-current injection test stand, and this problem can now be evaluated in great detail.

Studies were also made of the location and size of the three beam components entering the DCX-2 by using a target and intercepting the beam after the first half orbit. A surprising result discovered during these studies was that the optimum focal length of the magnetic lens was a function of the pulse rate of the beam. This problem will also be studied in greater detail with the 600-kv test stand.

Theory. — Further analysis of the stabilizing influence of an electron stream through the DCX-1 plasma suggests that, though instabilities at the lowest harmonics of the ion cyclotron frequency are stabilized, higher-frequency modes become unstable. Large energy transfer from ions to the electron stream, which was evidenced in recent experiments, even though low harmonic modes were partially stabilized, may be the result of these higher-frequency instabilities.

All electrostatic velocity-space instabilities have been interpreted as arising from the mechanism of the two-stream mode. Namely, charges streaming relative to potential peaks bunch in phase and add to the potential (destabilizing). The Penrose condition for electrostatic instability compares the sum of such responses to the stabilizing response of particles trapped in the potential valleys. The magnetic attraction of currents (a negative relativistic correction to the Coulomb force) induces just the opposite responses. The magnetic-pinch instability criterion may be regarded as a special case of the Penrose condition with e^2 replaced by its magnetic correction, $(-v^2/c^2)e^2$.

Radio-Frequency Heating. – Measurement of the transmission by the hot electron plasma of a beam of Li^+ ions has produced an indication that the plasma potential is probably several hundred volts positive. A possible reason for this positive potential is the necessity of the plasma to dispose of a large amount of cold positive ions produced at the plasma gas boundary. This cold plasma, which flows out along the magnetic field lines, constitutes some power drain but may also act to reduce electrostatic instabilities which would "dump" the hotter plasma. Work with the ELMO facility was interrupted by the failure of the smaller coil pair, which was being used to produce the mirror magnetic field for these experiments.

Experiments continued in the Physics Test Facility were on the effects of mixing xenon into the normal deuterium gas feeding the plasma. A xenon concentration of approximately 1 part in 20 in this mixture was sufficient to reduce the significant plasma parameters, that is, neutron flux, microwave noise, diamagnetism, and containment time, by factors of ~3 to 10. It is not surprising that a contaminant with high Z should act as a strong coolant for the electron gas. The fact that considerable plasma still exists is probably an indication that the high-Z atoms are confined to the plasma surface. As a consequence of their large ionization cross section (~50 times that of hydrogen for ~20-kev electrons) and low thermal velocity (~1/8 that of hydrogen), xenon atoms directed toward the plasma are ionized after only a short penetration. The Xe^+ ions would then be repelled by a positive plasma potential and move out along the magnetic lines of force.

Xenon concentration in the feed gas was correlated with the intensity of light emitted at the 3247-A wavelength of the Cu^0 resonance line. The presence of copper in the plasma volume has been noted previously and is presumably due to sputtering of the microwave cavity surface by impinging positive ions (General Mills Report 2309). With only deuterium as the plasma feed, this Cu^0 emission was barely observable. Upon adding xenon in concentrations of 5 to 30%, the intensity of the Cu^0 line increased linearly even after most plasma density and temperature indicators had suffered a marked decrease. Not until after xenon concentration reached 30% did the intensity of this Cu^0 emission finally begin to level off. No decrease in light intensity for this Cu^0 line was observed even at the maximum xenon pressure of 6×10^{-6} torr.

No characteristic emission was detected from either Cu^+ or Xe^{2+}. This observation is consistent with the suggestion that high-Z ions spend relatively short times in contact with the hot electrons.

The Physics Test Facility, with extra end pumping, has been employed in extensive tests to discover optimum methods of coupling microwave power from the klystron amplifiers to the plasma in the high-mode cavity. Results of these tests will facilitate the design of microwave components for the large-scale electron heating experiment mentioned in a previous report (ORNL-3388).

BIOLOGY AND MEDICINE PROGRAM

RADIATION EFFECTS

Pathology and Physiology. – Spleen cells from RF mice with radiogenic myeloid leukemia were serially passaged in irradiated and nonirradiated adult mice of the same strain. The incidence of takes increased in successive transplant generations, averaging about 70% after the twentieth passage. From mice with passaged leukemia, spleen cell suspensions centrifuged at 30,000 × g for 1 hr yielded supernatant fluids which transmitted the leukemia to ~50% of adult and ~60% of newborn recipients within 60 days after inoculation. Spontaneous myeloid leukemias have not been observed in control mice younger than ten months of age. Cell-free spleen filtrates from passaged leukemias induced the disease in ~3% of 310 unirradiated adult male recipients and in ~8% of 129 unirradiated newborn male recipients within 60 days. In 146 unirradiated newborn female recipients, cell-free filtrates induced thymic lymphomas as well as myeloid leukemias (~4% of each type within 120 days). On fractionation by zonal centrifugation in sucrose gradients, the leukemogenic activity in homogenized myeloid leukemic spleen cells sedimented in a manner appropriate for a virus particle of the type noted with the electron microscope in this leukemia [Cancer Res. 22, 728 (1962)]. Leukemogenic activity was also recovered from the blood plasma of leukemic donors. These results further implicate a viral factor in the pathogenesis of radiation-induced myeloid leukemia of the mouse.

Recovery from Somatic Effects of Radiation. – A number of codominant histocompatibility (H) factors affecting the fate of solid tumor or skin grafts in the mouse have been characterized genetically and immunologically. They have been thought by analogy to be the only factors determining the acceptance of grafted marrow, antibody-forming spleen cells, and leukemias, although it was known that in certain instances such cells would not grow after transplantation in F_1 hybrids as well as in syngeneic recipients. In this study it has been found that genetic factors, in addition to the well-known dominant H loci, are significant in determining histoincompatibilities specific for bone marrow grafts. Growth of marrow cell grafts was quantitatively assessed by determining their ability to incorporate I^{131}-deoxyuridine, a specific deoxyribonucleic acid precursor. The marrow recipients were (1) mice syngeneic with the donor strain (C57BL/10 ScSn), (2) F_1 hybrids from crosses between C57BL/10 mice with C3H DiSn or A/Sn partners, and (3) backcross progeny mice from matings of

F_1 hybrids to either parental strain. The genotype of the latter mice was determined for the H-2 locus by hemoagglutination, for the agouti (A) locus by coat color inspection, and for the hemoglobin locus by evaluation of peripheral blood hemoglobin characteristics. All the recipients were exposed to 700 r of whole-body x irradiation before marrow grafting. Seventy of such backcross progeny mice were tested for susceptibility or resistance to C57BL/10 marrow grafts. In 33 of them the marrow cells proliferated as well as in syngeneic recipients. In the remaining 37 backcross mice, the marrow cells failed to grow, as in F_1 hybrid recipients, suggesting that in the backcross progeny mice a single recessive gene affecting the take of C57BL/10 marrow grafts would segregate. This genetic factor, not expressed in heterozygotes, seemed to be linked with the H-2 locus; accordingly, no linkage could be detected with sex or with the genetic markers A or Hb. The mouse strains C3H DiSn and A/Sn do not share with C57BL/10 the recessive H factor, since their marrow cells grew equally well in syngeneic, F_1 hybrid, and backcross progeny mice.

Physiology of Blood Platelets. — It was previously shown that serum collected from thrombocytopenic rats (deficient in platelets) stimulated an increase in the number of circulating platelets upon injection into normal rats. Such serum was injected four times in a two-day period, and the maximum number of circulating platelets in the recipient was reached five days after initiating the injections. Serum-donor rats in these experiments had been made thrombocytopenic by massive bleeding or by injection of platelet-specific antiserum 24 hr before collection of the serum. It seemed likely that the serum of rats made thrombocytopenic by exposure to relatively large doses of whole-body radiation (LD_{50} within 30 days) would also contain a similar stimulatory activity at some time after irradiation, and that this stimulatory activity might play a part in the recovery of the platelet-producing mechanism of the bone marrow from radiation damage. Therefore, serum collected from rats 4, 6, 8, 10, 11, and 13 days after exposure to radiation was assayed by injection into normal recipients. However, no stimulatory activity was observed in any of these sera. It is possible that the titer of activity was low enough that it could not be detected by our relatively insensitive assay system, or, on the other hand, that the radiation may have inhibited the ability of the organism to produce the stimulatory material.

Medium(Low)-Level Long-Term Effects of Radiation. — Earlier observations have indicated that 24 hr after the injection of *Pseudomonas aeruginosa* into chronically irradiated mice, there is a secondary increase in the concentration of this organism in the liver. This increase is proportional to the accumulated exposure and is not seen in unirradiated mice. It has also been reported that the leakage of bacteria at the site of their intravenous inoculation results in a subcutaneous focus of infection in irradiated mice. The purpose of this study was to investigate the role of such a subcutaneous depot in the secondary increase of hepatic bacterial levels noted earlier. Young adult male CF-1 mice received an x-ray exposure of 50 r every 48 hr. At various accumulated exposures (500 to 2500 r), groups of mice were injected via tail vein with *P. aeruginosa*. Twenty-four hours after injection, the *Pseudomonas*

concentration was increased significantly above the control mean value both in the liver and at the inoculation site in the tail. The tail values were usually higher. All the irradiated mice were leukopenic, and Pseudomonas could frequently be cultured from their blood. RF mice treated similarly developed the secondary increase in Pseudomonas in the liver only after higher accumulated x-ray exposures, although the increase was marked after 1000 r given in a single exposure. These results indicate the importance of sources other than the liver itself in the secondary rise of hepatic bacterial concentrations in irradiated mice.

COMBATING DETRIMENTAL EFFECTS OF RADIATION

Mammalian Recovery. — Iron-59 uptake by spleens of lethally x-irradiated mice is a function of the number of isologous bone marrow cells injected. Uptake depends on erythropoietic activity in the spleen, which reflects quantitative transplantation of marrow cells. This system was developed as a means of evaluating in vitro treatment of cells injured by radiation. $(C3H \times C57BL)F_1$ mice are exposed to 900 r and injected with isologous bone marrow. Eight days after marrow injection, Fe^{59} is given intraperitoneally and spleen is removed 6 hr later. Uptake is linear with marrow dose between 0.05×10^6 and 0.8×10^6 cells. Beyond 0.8×10^6 cells, reduction of uptake was observed rather than the expected plateau. Spleen weight is also reduced after high marrow doses. These decreases may represent a physiologic response that signals a shutdown in cell proliferation in the hyperactive spleen which, in mice given 0.8×10^6 cells, takes up four times as much iron as does the spleen of a normal mouse. Short-term culture conditions were investigated to obtain an optimal environment for irradiated marrow cells. Initially, Tyrode's solution was used for a 6-hr incubation period, noting both iron uptake of spleen induced by these cells and cell counts in the incubated cultures. Up to 50% variation was observed not only in erythropoietic activity of cells, but also in the number of cells remaining after 6 hr of incubation at 23°C. Variation has been reduced by use of 10% calf serum in Tyrode's solution without calcium. Reduction in cell number (10 to 15%) still is observed, but reduction in erythropoietic activity is minimized.

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