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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

MAY 1961

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Oak Ridge, Tennessee
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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

MAY 1961

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

REACTOR DEVELOPMENT PROGRAM

GAS-COOLED REACTOR PROJECT

Irradiation Program. – Longitudinal sections of the type 304 stainless steel cladding of ORR-irradiated capsules 02-2, 05-2, 06-2, and 08-2 were examined metallographically. The end-cap welds were intact, and no deleterious effects of irradiation were evident. At cladding positions adjacent to the UO_2 pellet interfaces, however, two conditions were noted that were not present in the unirradiated control samples. In all the irradiated samples, small voids were seen in the as-polished condition. Relatively large voids (~0.003 in. long) were evident at a cladding position where a pellet interface "wrinkle" was observed. The other change in microstructure noted was the formation of a second phase in the grain boundaries. This phase has been tentatively identified as sigma by both metallographic and x-ray examinations.

Examinations of three LITR-irradiated capsules containing UC fuel material revealed reactions between the UC and the tantalum can. Gross damage of the outer Inconel capsules also occurred during irradiation of the second and third capsules, at central temperatures in the range 2000 to 2500°F, to approximate burnups of 11,000 to 8000 Mwd/metric ton respectively. Analysis of gas collected from the first capsule, which was irradiated to a burnup of approximately 4000 Mwd/metric ton, showed that 1.7% of the Kr^{85} generated by fission was released from the UC.

The release of short-lived gases, especially Xe^{138} and Kr^{88}, from irradiated UO_2 was studied in instantaneous fission-gas-release experiments. These gases reach equilibrium release within 24 hr, and the temperature dependence of release can be established within a few days. The diffusion coefficients obtained for Kr^{88} over a four-month period of irradiation can be separated into two groups: (1) the values obtained before the UO_2 sample was operated at its highest temperature (1100°C) for ten days and (2) the values obtained after the high-temperature operation. The gas retention of the UO_2 was decreased by the high-temperature operation.

The eight group III capsules being irradiated in the ORR were withdrawn on May 8, 1961, after eight months of irradiation, and eight group

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IV capsules were inserted. The group IV capsules include two stainless-steel-clad capsules containing UO_2 pellets of different grain sizes, two similar capsules containing swaged UO_2, and two containing tamp-packed UO_2. The remaining two capsules consist of beryllium-clad UO_2 pellets supplied by the French CEA. One capsule has an electron-beam-welded closure, and the other has a hand-welded closure.

Also installed in the ORR irradiation facility during this shutdown were new mounting and positioning brackets and two new base plates containing mechanisms which provide for manual adjustment of each capsule within a zone 1 to 8.9 in. from the west wall of the reactor while the reactor is operating. This unit will be used to compensate for changes in neutron flux levels. New gas circulators were incorporated in the control-gas circuits of each of the eight capsules. The new circulators differ from those used previously only in that the pump speed has been increased from 500 to 1000 rpm. The higher speed should prevent the copper-Teflon vanes from sticking and causing temperature excursions such as those which occurred previously. Two thermal-neutron-monitor thermopiles were installed 22-1/2 in. west of the reactor pool-side windows at the midpoint behind each of the north and south capsule groups.

The in-pile and in-pool piping of GCR-ORR loop No. 1 was completed. This piping is required to provide double containment and to assure more efficient flow of the helium coolant. The loop performed satisfactorily in a trial run with the reactor operating at 5 Mw. The test specimens installed in the loop consist of approximately 30 pieces of weld metal representative of that being used in the main GCR containment pressure vessel. This steel will be irradiated at approximately 550°F for at least one seven-week cycle to study the embrittling effects of irradiation.

Reactor Physics. – The first results were obtained in a study of the power distribution within 19-rod cluster fuel elements in the EGCR core. For a cluster with the EGCR enrichment (2.46%) and very nearly the same volume of fuel, the ratios of the rod power to the average power per rod for the central rod and for rods of the first and second rings are, respectively, 0.840, 0.925, and 1.050. The power density across the outer ring of fuel rods drops by a factor of 1.05, and that across the first ring drops by a factor of 1.06.

A series of calculations is in progress to determine the reactivity lifetime of fuel elements consisting initially of U^{235}, thorium, and graphite. The specified conditions were a moderator temperature of 1000°K, core leakage of 5%, fluxes characteristic of an equilibrium fuel cycle, and an excess k of 1%. The fuel specific power and the composition of fresh fuel elements were varied. The fission product cross sections of Nephew (ORNL-2869) were used both for thermal and resonance absorption.

The atomic ratio of carbon to uranium in the fresh fuel was varied from 2000 to 8000 with only minor (10–15%) changes in lifetime. A maximum lifetime was obtained with a carbon-to-uranium ratio of about 4000. As the ratio was increased, the absorptions in graphite became excessive; as it was decreased, the fission product absorptions became excessive and the value of $\bar{\eta}$ decreased. The optimum (for lifetime) atomic ratio of thorium to uranium in the fresh fuel was found to vary from about 7 to 11 as the specific power was decreased from 10,000 to 500 kw/kg. Some of the lifetimes obtained with optimum fuel compositions are:

Specific Power (kw/kg)	Fuel Lifetime (fissions per initial fissile atom)
500	2.2
1,000	2.0
5,000	1.6
10,000	1.4

The specific power is an important variable in that it determines the amounts of xenon poisoning, required control-rod worth for samarium override, and protactinium poisoning.

EGCR Control-Rod Mockup Tests. – The EGCR control-rod mockup was assembled, and coolant-flow and pressure-drop tests were performed. The pressure-drop measurements confirmed the calculated values; however, the joint leakage was less than expected. Also, the rod weight of 137-3/4 lb was near the computed value of 138 lb. One questionable feature of the design was apparent in that the rod segments would not stay centered on the central support rod. The control rod was therefore modified by reducing the clearance between the central rod and the internal spacers and by inserting one additional spacer just above each joint. This modification allows the segments to be aligned while maintaining sufficient rod flexibility. Bids for manufacture of the control rods have been requested, and about 16 vendors are expected to submit bids by June 6, 1961.

Graphite Outgassing Studies. – Specimens machined from block No. 4 (file No. 3) of the EGCR graphite were degassed by external resistance heating in the temperature range 30 to 1000°C. The specimens had been machined in different sizes (1-in. diameter, 1-in. length; 1-in. diameter, 4-in. length; and 2-1/4-in. diameter, 2-in. length) from the central region of a 6-in.-thick slab cut perpendicular to the extrusion axis of the 20-ft bar. The degassing behavior of all the samples was similar. Significant amounts of SO_2 were released by all specimens during heating from 300 to 600°C. The volumes of gas evolved as a function of time at 300, 600, and 1000°C for the various specimens indicated that the effect of the geometry of the specimen on the rate of release of the gas was unimportant. Variations found from specimen to specimen of the same geometry were as large as those observed for specimens of different geometry. An increase in rate of release with increasing time of degassing was observed in all cases at 1000°C.

Adsorbers for Removing Fission Product Gases from Coolants. – Studies on the removal of radioactive iodine vapor from high-temperature helium streams continued, with emphasis on iodine retention by Whetlerite charcoal (Pittsburgh BPL charcoal impregnated with salts of silver, chromium, and copper). The migration rate of iodine through relatively deep beds (~4 in. of Whetlerite charcoal, 12/30 mesh) is being studied as a function of temperature. The second test of the series was terminated after 897 hr at 425°C with a helium velocity of 53 fpm. The iodine was contained in the first 0.75 in. of charcoal, and the over-all iodine migration was

no more than 0.07 in. Similar results were obtained from the first test at 325°C.

Evaluation of ICI Beryllium Tubing. – Approximately 200 ft of extruded beryllium tubing (0.300-in.-ID, 0.040-in.-wall) was received from Imperial Chemical Industries Limited for nondestructive evaluation. Low-voltage radiographic examinations of the entire shipment were completed. Numerous, fine, high-density particles were detected throughout the tubing. In addition, a number of extrusion marks of varying severity and a few isolated pits were noted. Fluorescent-penetrant evaluation has, to date, disclosed only shallow pinholes. Ultrasonic examinations are not complete, but it is evident that some of the tubing will be rejected for failure to meet the prescribed wall-thickness tolerance.

Coated-Particle Fuel Element Development. – Preirradiation evaluation was completed for spheroidal uranium carbide particles coated with pyrolytically deposited carbon, sample 3M, lot 10695-45. The surface contamination after three thermal cycles to approximately 1400°C had increased to 0.003% from an as-received surface contamination of 0.0009%. The amount of uranium dissolved by acid leaching after thermal cycling was 0.03% of the total uranium initially present in the particles, indicating that some of the particles were damaged. An effort will be made to evaluate this material more completely to determine the cause of the poor thermal cycling resistance.

Fuel capsules were assembled for the second LITR irradiation experiment. Coated-particle sample 3M-SP-2 was selected for this experiment. The capsules are similar to those used in the first LITR experiment (L-CPlab) except that the ends of the tantalum cans have been modified to facilitate opening in the hot cells.

Two fission-gas-retention tests were performed on 3M pyrolytic-carbon-coated UO_2 particles. The first was a 140-hr isothermal test at 1960°C. No Xe^{133} was released; however, activity was found deposited on the furnace. This activity was identified as Ba^{140}-La^{140}, almost in equilibrium ratio.

The second test was for 22 hr at 1900°C. Again no Xe^{133} was released. However, activity was found on the tantalum wire on which the crucible was hung in the furnace. In the first 1 in. of the wire, Ru^{103} was found; in the next 1 in., there was a small amount of Ru^{103} and gross amounts of equilibrium Ba^{140}-La^{140}. The rest of the wire (about 5 in.) was virtually clean, although small amounts of Ba^{140}-La^{140} were found toward the colder sections of the wire. No other fission product gamma activity was observed.

THERMAL-BREEDER REACTOR PROGRAM

Homogeneous Reactor Program

Homogeneous Reactor Test. – The experimental program of the HRT was terminated late in April, and the reactor was shut down permanently as scheduled by May 1.

During the final run (No. 25), mixing of fuel between the core and blanket regions increased sharply (reported in April). After shutdown an inspection of the interior of the core revealed that the upper Zircaloy vessel patch had become detached from the vessel and was lying on the upper diffuser screen along with a portion of the bolt which had held it in place. The lower patch was in place, but its gold gasket was loose.

The detached upper patch and bolt fragment were removed from the core for examination. Preliminary inspection revealed that the bolt had melted near the toggle nut about 1/4 in. from the patch on the blanket side. There was no appreciable attack on the core side of the patch or on its surfaces which contacted the vessel or bolt. The melting of the bolt was attributed to boiling and collection of uranium under the adverse flow conditions existing in the blanket.

Operations necessary for removal of corrosion specimens and for inspection and storage of the reactor (in an assembled state) are now in progress.

Thorium Blanket Studies. – The investigation of the application of the sol-gel process to the preparation of thoria pellets suitable for use as blanket material in an aqueous homogeneous reactor was continued. Eight materials prepared by the sol-gel process with varying N/Th ratios, along with a material prepared by peroxide precipitation, were screened before and after grinding in a spouted bed for 15 hr. Of these preparations, four had > 50% survival of rounded particles > 10 mesh. These included the peroxide-precipitated material and the three sol-gel materials with N/Th $\leq$ 0.11. The peroxide-precipitated material had crystallites, at the sol stage, nearly twice the average for sol-gel materials used. From this result it was concluded that lower nitrate concentration and larger crystallite size for the precursor oxide are important for obtaining strong particles by the sol-gel process.

Reactor Analysis. – Power-trace data from the HRT were subjected to a variety of statistical tests covering both the observed fluctuations and the reactivity changes necessary to produce them. The positive power and reactivity deviations were found to be consistently larger on the average than the negative deviations. The percentage deviation increased with core power level. It was not established whether pressure, temperature, or flow rate affected the deviations. Comparison of calculated fluid recirculation rates with measurements made in a core mockup showed a strong similarity. While agreement between the mockup measurements and the reactor data is not complete, the comparison offers considerable support to the idea that hydrodynamic fluctuations were a major cause of the power deviations.

On changing the power level in the HRT, temperature transients due to density changes may occur. It was calculated that going from zero power to 5 Mw produces an initial temperature rise of about 0.7°C followed by a gradual decrease over a 2-hr period to an equilibrium point 1.3°C below the initial assumed level of 265°C.

Homogeneous Reactor Instrumentation. – The HRT instruments are being placed in a stand-by condition. Instruments will be left in place wherever this is practical and will be secured in such a manner as to prevent tampering or deterioration. Electrical circuits will be de-energized

wherever possible, and the air supply system will be secured. Precautions are being taken to minimize condensation in the air lines and instruments. Stand-by monitoring devices will be provided as required for personnel safety and equipment protection. A telemetering system is being installed to transmit alarm signals to the ORNL Guard Headquarters.

Metallurgy and Ceramics. – A study of several pellet-sintering methods was made which led to the development of a successful procedure for fabricating ThO_2–1% UO_2 spheres. In this procedure, cubes are tumbled into spheres by use of techniques described in ORNL-3120. After rough tumbling, the spheres are fired in air at 600°C to remove Carbowax binder and are then fired in H_2 at 1350°C. After wet tumbling to polish the surfaces, they are fired in hydrogen at 1800°C to produce a final density of 9.45 g/cm^3.

One kilogram of ThO_2–1% UO_2 spheres was prepared by this method, and the pellets appear visually to be sound and crack-free. These pellets will be tested for uranium leaching characteristics.

Out-of-Pile Blanket Materials Tests. – A study was made for determining the susceptibility of Zircaloy-2 and other zirconium-base alloys to hydriding during exposure to flowing slurries of thoria and of Th-U oxides at temperatures of 200, 280, and 335°C. In one phase of the study, with toroids in which pin-type specimens of the alloys were subjected to slug flow of slurry at 26 fps, oxygen overpressure inhibited hydriding of all alloys studied. On the other hand, when hydrogen and hydrogen-argon mixtures were used, the rate and extent of hydriding of the alloys increased with increases in gas overpressures in the range 15 to 260 psig (room temperature). For any given alloy in a specific type of slurry for a specific test time, there was a threshold hydrogen overpressure above which accelerated hydride formation resulted. Although threshold pressures varied markedly with different slurry preparations, in general the threshold pressure was lowered as the percentage of uranium in the oxides increased up to about 20 wt % U with respect to thorium. For a given system the extent of hydriding was proportional to test duration.

At different temperatures in comparable tests the susceptibility of the alloys to hydriding was greatest at 280°C, moderate at 200°C, and not detectable at 335°C.

Where severe hydriding occurred, it resulted from the reaction of the alloys with molecular hydrogen from the overpressurizing gas as well as hydrogen which might have resulted from corrosion.

The alloys tested were, in their order of decreasing susceptibility to hydriding: Zircaloy-2 (coarse grained), Zircaloy-2 (fine grained), Zircaloy-3, and Zircaloy-4.

Based on metallographic examinations of over 200 specimens, uptake of hydrogen by the alloys was initiated at the surface of the specimens at areas where protective oxide films were continually damaged or removed due to abrasion by impinging slurry particles. Hydriding progressed by intergranular diffusion of hydrogen into the alloys. In many specimens which were grossly hydrided, additional paths for entrance of hydrogen were available through cracks and fissures which resulted from the volume expansion associated with zirconium hydride formation.

In-Pile Blanket Materials Tests. – Direct observation by electron micrography of samples of solids which had been circulated in-pile in the

slurry loop experiment showed successively advanced stages of particle degradation as irradiation and circulation continued. Initial in-pile samples showed a rounding of sharp edges of particles. The progressively increasing production of fine particles after about 400 hr of irradiation was readily apparent in the photographs.

The total burnup of U^{235} in the in-pile slurry loop experiment was estimated at 0.92% of the original 0.4% U (with respect to thorium), with a total exposure of 1.6×10^{19} nvt. The flux averaged over the entire volume of the main stream was 2.4×10^{12} nv. Approximately 7×10^{16} fissions per gram of solids were obtained. Analyses of Cs^{137}, Zr^{95}, Ce^{95}, and Pa^{233} in both the solid and liquid phases indicated that only Cs^{137} appeared in appreciable concentrations in the liquid phase. The distribution ratio (solid/liquid) was found to be about unity for Cs^{137}, compared with values of over 10^4 for the other nuclides.

In postirradiation examination of the loop after it was sectioned, direct observation of the internal surface of the piping at the inlet and outlet of the high-flux region of the loop showed only insignificant amounts of dispersed material remaining on the piping. Similarly, corrosion coupons removed from specimen holders in the core section showed no marked accumulation of solids. Weights of solids removed from coupons by scrubbing ranged from 1 to 3 mg/cm^2. Weight gains for scrubbed Zircaloy-2 specimens (relative to original weights) ranged up to 2 mg/cm^2. A loss of 6 mg/cm^2 of metal, or a pickup of 2 mg/cm^2 of oxygen, would correspond to a generalized corrosion rate of 1 mil/yr. Zircaloy-2 specimens located at entrance regions of specimen holders showed weight losses, particularly for the higher velocity (22-fps) holder.

The autoclave from experiment L5Z-155S was opened, and the slurry was removed for analyses. This experiment had been performed to evaluate the radiation effect on the degradation of particles gently suspended in a rocking autoclave in comparison with that observed in the slurry pumped in the in-pile slurry loop experiment L-2-27S. In the autoclave experiment the thermal-neutron dose was 1.1×10^{19} nvt; in the loop experiment the dose was 1.6×10^{19} nvt. The Zircaloy-2 autoclave contained a thoria-urania slurry (0.4% U based on thorium) which was a replica of that used in the in-pile slurry loop experiment. The same catalyst and thoria-urania (batch DT-22) preparations were used.

The surface area of the original (batch DT-22) thoria-urania (92%) was 2 m^2/g; that of the finely divided palladium-thoria catalyst (8%) was 53 m^2/g. The surface area of the unirradiated mixture was 8 m^2/g. After irradiation, the surface area of the slurry was 30 m^2/g.

Particle-size analyses of two samples of the irradiated slurry compared with an unirradiated control sample indicated no degradation of the slurry particles. From the results of the physical analyses so far, it is inferred that in-pile irradiation does indeed cause defects in the slurry particles, as observed by an increase in the surface area, but that particle agitation more vigorous than that experienced in a rocking autoclave is necessary to actually break up the slurry particles.

Evaluation Studies

Thorium-Breeder-Reactor Evaluation Studies. — The fuel costs in a CANDU-type reactor fueled with Th^{232} and uranium highly enriched with U^{235}

(CANadian Deuterium Uranium THorium) were estimated by using cost bases and ground rules used in the evaluation of five thermal breeder reactors. The costs obtained, based on a "throwaway" CANDUTH cycle, were about the same as those obtained for CANDU.

Preliminary nuclear calculations for a large one-region molten-salt reactor indicate that even with a throwaway cycle it may be possible to achieve breeding ratios greater than unity. However, the fuel costs for this cycle in which salt and thorium are discarded, but U^{233} is recovered, will be about twice as high as the costs of a two-region, two-fluid system (MSBR), but may be below 2 mills/kwhr if 1000 Mw (electrical) can be obtained from a single 20-ft-diam core.

In studies of a gas-cooled, graphite-moderated breeder reactor (GGBR) reported previously, it was assumed that none of the xenon produced in the fuel matrix would escape and that a neutron loss corresponding to about 0.06 in breeding ratio would be incurred. The use of AGOT-grade graphite, however, may permit xenon to diffuse rapidly into the gas stream, resulting in little xenon poisoning. A neutron saving of this magnitude could possibly make the GGBR competitive with the molten-salt reactor as a breeder.

MOLTEN-SALT REACTOR PROJECT

MSRE Design. – Piping designs were completed for the fuel- and coolant-salt systems, and the pump-support arrangement was designed. Locations were assigned for electrical disconnects for heaters and thermocouple disconnects within the reactor cell.

Detailing of the reactor-vessel design was completed, except for the 10-in. access port. The access port is being designed, as are the control-rod penetrations which are a part of this port.

Reactor-cell penetrations and the numerous flow diagrams are being detailed.

The pipe-heater design is also being detailed. A heater design for the pump, which is one of the most difficult components to heat, was laid out in rough form.

Drafting continued for the lower shielding plugs, the maintenance control room, the electrical-service rooms, and the radiator support. Drawings of drain-tank-pit reinforcing are 90% complete.

The location of the reactor-cell exhaust duct was changed slightly, which will require a reworking of some drawings. Drawings of the storage-pit ventilation system were finished. Electrical-distribution drawings are progressing; they include power, auxiliary power, emergency power, and lighting in control rooms, cells, and crane-bay areas.

The layout of the maintenance control room was revised to accommodate additional equipment. The design for a 1500-w portable light unit to provide additional lighting for television viewing was accepted.

The shielding-window drawings were revised to comply with comments received on the preliminary drawings and with production tolerances.

The design of the freeze-flange torqueing tool was made, and drawings are in progress for extension wrenches for this tool.

Component Development. – The final design of the full-scale hydraulic model of the MSRE core with its associated loop was completed, and fabrication was started.

Experiments conducted on the one-fifth-scale model to determine the effect of the flow on solids indicate that some solids, with densities as high as that of UO_2 and diameters as small as 20 μ, will settle in the lower head of the core.

Operation of the engineering test loop continues with the MSRE coolant salt. The loop is operating isothermally at 1100°F with a flow rate of 50 gpm. Salt samples removed during operation indicated an oxide content of only 300 ppm in solution.

Testing of the fuel-salt-sample capsule indicated that the copper capsule must be destroyed to remove the sample. A capsule with a thin wall which can be easily crushed was designed.

The reactor-vessel heater test operated satisfactorily for 11 weeks with no obvious heater deterioration. A system for removing heat from the reactor side of the test was used to evaluate permissible heat rates into the reactor vessel during the initial heatup. It was found that at 1250°F, 4.3 kw could be added to the reactor vessel above that required to overcome the heat loss.

Efforts to incorporate conical gasket-type gas seals in the MSRE freeze flange were stopped, because studies have indicated that the critical alignment requirement during remote assembly is prohibitively complex. The efforts are being directed toward ring-joint seals, which are more compatible with remote assembly.

Remote Maintenance. – Test brazes on a tentative design for a 1-1/2-in. pipe joint pointed up the alignment problem in maintaining the correct gap for braze-metal flow. Since remote preparation and assembly of very close clearance joints is undesirable, a design with a 50-mil/in. taper to permit assembly with no gap is being constructed for testing.

The MSRE scale model is being revised to reflect the new layout in the reactor and drain-tank areas.

Construction of the equipment mockup was begun; the initial test will be on the remote operability of the freeze flange.

Layout design studies were started for a portable sliding shield for use in the maintenance of reactor-cell equipment.

MSRE Instrumentation. – Preparation of instrument application diagrams is continuing. Eight drawings were issued for comment. Six of these drawings had previously been issued for comment and had been revised to incorporate recommended changes and a revised instrument numbering system. Preliminary work began on four additional diagrams. Present estimates are that 14 application diagrams will be required to cover all instrumentation on the project.

Development work on thermocouple and electrical disconnects was completed, and a specification for procurement of the electrical disconnects was prepared.

Investigation of requirements for the data-handling system is continuing. Tabulation of inputs to the data system is nearing completion. The feasibility of using a differential scan alarm system, for scanning pipe and vessel temperatures during reactor startup, is being investigated.

Location of in-cell instrumentation is being incorporated in arrangement drawings of the primary containment vessel.

A quarter-scale mockup of the main control board was completed. This mockup, which was a first approach to the problem of panel-board layout, is a fully graphic board. A semigraphic board will be designed and proposed as an alternative.

Pump Development. – The test pump with a molten-salt-lubricated journal bearing has operated continuously for 9630 hr with $LiF-BeF_2-UF_4$ (62-37-1 mole %) at 1225°F. The pump has been started and stopped 78 times.

The Inconel centrifugal pump (PKP type) was shut down because of a mechanical-electrical failure in the drive motor after 5436 hr of continuous operation with $LiF-BeF_2-ThF_4-UF_4$ (65-30-4-1 mole %) at 1225°F, 1950 rpm, and 510 gpm. A sweep-gas flow rate of 2000 cm^3/min down the pump-shaft annulus provided a dilution factor of 1100 against the back diffusion of Kr^{85} from the pump-tank gas space, subsequent to the preliminary testing previously reported.

Water testing of the hydraulic configuration for the prototype fuel pump was completed satisfactorily. Water testing of the hydraulic configuration of the coolant pump was started.

The foundry was provided with new composition limits for INOR-8, which should increase the fluidity of the casting metal and aid in producing the impeller and volute castings for the MSRE pumps. Fabrication of components for the hot-test stand for the prototype fuel pump was restarted after one month's suspension.

Debugging of a stress-analysis Oracle code for the pump tank continues. Presently, the code satisfactorily computes pressure and load stresses but not the thermal stresses. Computations of thermal stresses in the cylindrical portion of the pump tank and in the spherical shell are in progress.

Procurement and Construction. – Requests for bids on INOR-8 material, from which the MSRE is to be constructed, were sent to prospective vendors. Purchase contracts for this material will be executed in June, and delivery of the plate and bar is expected by September. Delivery of the pipe and tubing is scheduled for December.

Demolition and minor alteration work was started in Building 7503, the MSRE site. This work, being performed by the H. K. Ferguson Company on a cost-plus-fixed-fee basis, consists in the removal of old shielding walls, excavation of the drain-tank pit, construction of an additional stairway, and some minor electrical work. The work is approximately 60% complete; it will be completed by June 15.

Chemistry. – The accumulation of corrosion products in molten fluoride fuels has been monitored by periodic sampling during long-term corrosion tests involving nickel alloys (INOR-8 and Inconel). Recently collected chemical analyses now provide complete life histories for a group of pumped loops which completed operation several months ago. In agreement with earlier results, and because Cr^0 is the most active element in the alloys, Cr^{2+} is the predominant corrosion product and provides the important indications of significant trends with time. The best example of the expected favorable behavior is still an INOR-8 system in which $LiF-BeF_2-ThF_4-UF_4$ (62.0-36.5-1.0-0.5 mole %) was circulated for 14,580 hr at

700°C and the Cr^{2+} concentration remained steady at 550 ppm. Such results have now been confirmed with a fuel having a high ThF_4 content, $LiF-BeF_2-ThF_4-UF_4$ (67-18.5-14.0-0.5 mole %), which circulated in INOR-8 at 700°C for 10,000 hr and developed a Cr^{2+} concentration of 500 ppm. The effect of temperature was shown by a similar system which manifested a steady-state Cr^{2+} concentration of 700 ppm after 10,800 hr at 760°C. The influence of the concentration of UF_4, the main oxidizing agent in the fuel, was demonstrated with $LiF-BeF_2-UF_4$ (70-10-20 mole %). As previously reported, the Cr^{2+} concentration developed with 20 mole % UF_4 had reached 1700 ppm in 8200 hr and showed no sign of leveling off.

Metallurgy. — A program is continuing for studying the crack susceptibility of various heats of INOR-8 as a result of welding. One-inch plates from seven heats of material were welded and were tested in bend tests and examined metallographically. Results verified the prediction that weld cracking was associated with inadequate purification and deoxidation of the melt during casting. Crack-free weldments were made from heats of both vacuum-melted and air-melted INOR-8 with improved deoxidation practices. Acceptable heats have now been made by both major INOR-8 manufacturers.

Specification of a weldability test method that is acceptable to plate manufacturers was prepared for inclusion in the procurement specifications for INOR-8. This method includes the restrained, 1-in.-thick weld test.

Corrosion Tests. — Metallographic examination of specimens from INOR-8 forced-circulation loop MSRP-6, which operated for 20,000 hr under the conditions given below, revealed only light surface roughening and the formation of a discontinuous surface layer. The surface layer occurred only in the hot-leg regions and varied in thickness to a maximum of 2 mils.

Salt mixture	$LiF-BeF_2-UF_4-ThF_4$ (62-36.5-0.5-1.0 mole %)
Maximum salt-metal interface temperature	1300°F
Maximum bulk-salt temperature	1250°F
Loop ΔT	200°F
Flow rate	15 gpm
Reynolds number	2300

In-Pile Tests. — The third MSRE graphite-fuel capsule assembly (ORNL-MTR-47-3) was inserted in the MTR during the week of May 1. The experiment is operating satisfactorily except that the capsule graphite temperatures are low. The graphite-to-salt interface temperatures of the unimpregnated capsules are about 1430°F, or 120°F below design. Those of the two impregnated capsules are 1500 and 1640°F, or 300 and 140°F below design. These temperatures are well above the maximum MSRE core temperature of 1275°F, and the operating conditions are considered to be satisfactory. The cause of the low temperatures is not certain. The flux distribution in the MTR may have been shifted as a result of the recent removal of the

ANP experiments and a change in control-rod location from one distant to this experiment to one near by. The helium-gas gaps in the experiment, which provide the thermal resistance, may be slightly small.

Reactor Analysis. – A summary of the various radiation source strengths in the MSRE equipment cells was prepared to aid in determining estimates of cell radiation levels. The residual activity in the heat exchanger and pump bowl following 10-Mw operation for one year was estimated and included in the summary.

Assuming that 100% of the fission products considered were plated out in the heat exchanger, the resulting gamma emission rate at 10 and 30 days after shutdown corresponded to 2.8 and 1.9 kw respectively.

If all the daughter products of xenon and zirconium plated out in the pump bowl, the gamma activity at 10 and 30 days' cooling time would correspond to 0.13 and 0.056 kw respectively.

HIGH FLUX ISOTOPE REACTOR

Physics. – The reactivity worth of the HFIR control plates was recently calculated by using a one-dimensional, transport-theory reactor code (DSN). By use of four-group cross sections that were derived primarily from a 34-group diffusion theory calculation (GNU), the worth (k_{eff} – 1) of the 1/2-in.-thick gray region (nickel) and of the 1/4-in.-thick black region (0.030 in. nickel plus 0.220 in. silver) was calculated to be 0.10 and 0.18 respectively. These calculations treated the various narrow regions of plate and coolant as discrete regions; corresponding values for a homogenized control region (3 cm thick) were, respectively, 0.13 and 0.23. Diffusion theory (GNU) gave 0.13 and 0.26 for the latter values of control-plate worth.

Further calculations have been made in connection with xenon and samarium poisoning problems, with low-power samarium burnup being considered for a core that has been shut down for several days. Based on a 15-day normal operating lifetime, a core that has been shut down for a prolonged period after no less than about 11-1/2 days cannot be restarted without some samarium being burned out or fuel added.

Until the core is approximately 13 days old, sufficient samarium can be burned out at low power (approximately 1 Mw) to permit the core to complete and even exceed by about one-half day the normal fuel cycle at full power. Shutdown after 13 days plus samarium burnup results in a fuel cycle at full power shorter than normal.

Assuming that low-power samarium burnup would be accomplished in an auxiliary low-power facility in which heat is transferred by natural convection, and considering the redistribution of power resulting from redistribution of the poisons and repositioning of the control plates, it was estimated that the maximum achievable power level without film boiling at the hot spot would be about 1 Mw. Under these conditions the time required to adequately burn the samarium ranged from zero for an 11-1/2-day-old core to about 50 days for a 13-day core. For a 13-1/2-day core the required burning time was about 100 days.

Control and Instrumentation. – A laminate of air, water, and iron has been proposed for the ion-chamber neutron collimator located between the

beryllium and the pressure-vessel wall. Calculations are now in progress to determine the resulting increase in neutron current at the chamber location and to determine the off-axis "noise" component of this current.

A system for the on-the-line testing of the inlet and outlet temperature sensors for the coincidence safety system was developed. A prototype injection nozzle was fabricated for test in the mockup. This prototype will be employed to determine the location of the nozzle with respect to the sensors and the Δp and Δt between the injected water and the process water.

A gravity-flow trough will be used to conduct a sample of all cooling-tower water as it comes from the tower to a common temperature element, which will be used to control the secondary cooling-water temperature. Analog studies of the secondary cooling-water system indicate that adequate control will be obtained.

Corrosion. – Several heat-flux corrosion tests were conducted to complete the corrosion program in support of the HFIR. The following paragraphs summarize the effect of heat flux, pH, and coolant temperature on the corrosion of 6061 aluminum by water.

Ten-day tests were completed at heat fluxes of 1.0, 1.5, and 2.0×10^6 Btu hr^{-1} ft^{-2}. In all tests the flow rate was 40 fps, the pressure was 900 psi, the pH of the coolant was 5.0, and the inlet water temperature was adjusted so that the outlet water temperature was 220°F. The maximum rates of temperature increase were: 2, 6, and 17°F/day for respective heat fluxes of 1.0×10^6, 1.5×10^6, and 2.0×10^6 Btu hr^{-1} ft^{-2}.

A series of tests was conducted to determine the effect of pH in the range of 5.0 to 6.4 (HNO_3). The test conditions were: heat flux, 2.0×10^6 Btu hr^{-1} ft^{-2}; flow rate, 40 fps; coolant temperature, 176 to 220°F; pressure, 900 psi. The maximum rates of temperature increase were 17°F/day at pH values of 5.0 and 5.3, 31°F/day at a pH of 5.7, and 48°F/day at a pH of 6.4 (deionized water). In the last two cases, film stripping and localized attack of the aluminum occurred.

Increasing the coolant temperature increased the rate of oxide formation. For example, at a heat flux of 1.5×10^6 Btu hr^{-1} ft^{-2}, the rate of temperature increase was 5 to 6°F per day when the outlet water temperature was 220°F and 12 to 14°F per day when it was 250°F.

The data indicate that at a fixed pH and at flow rates, coolant temperatures, and heat fluxes of interest to the HFIR, the rate of oxide formation (and presumably corrosion) is determined to a large extent by the specimen temperature.

Examination of the corrosion specimens is not complete, but except for those cases where film stripping occurred, attack of the aluminum was uniform and never exceeded 2 mils penetration during ten-day tests. In those cases where film stripping occurred, localized attack to a depth of 5 to 10 mils was observed. Under the most severe conditions expected during operation of the HFIR, penetration of the aluminum cladding is estimated to be about 1 mil per ten days, provided that the pH of the water is maintained between 5.0 and 5.3.

Metallurgy. – The method of correlating uranium concentration with measured densities in the HFIR alloy cores was found to be accurate to approximately ±0.5%. This was observed in fuel cores of 24 wt % U selected from those made for the critical test element, as well as in a study of alloy castings ranging in composition from 18 to 30 wt % U.

Analysis of the data accumulated during the rolling of plates for the critical element was completed for the outer annulus. The widths of the "meat" in the plates were found to vary by approximately ±1/16 in. A taper in the width was observed at the ends of the plates for approximately 1 in. of length. Lengths varied by ±1/2 in. A metallographic examination of three plates indicated that the thickness dimensions were well within the specified values.

Spacing measurements were obtained for inner-annulus water channels of the critical fuel element. In those locations where Teflon spacer strips were used during welding, a fair control was observed on dimensions.

Design. – As a result of discussions with machine-shop personnel, the permanent beryllium reflector concept was modified to reduce the estimated machining costs. In the revised concept, coolant is supplied from a common plenum chamber to both the removable and permanent reflector region, with flow control obtained by directing flow through an orifice into the common plenum. The outer ring of the previous removable-reflector design was incorporated into the permanent reflector in the form of a segmented, semipermanent ring. Four of the eight segments of this ring may be removed to permit removal and replacement of the control plates. The other four segments may also be removed, but with some difficulty, in the event that they require replacement because of radiation damage. The engineering facility tubes were relocated to a point about 2 in. nearer the core center line by grooving the outer cylindrical surface of the permanent reflector. The horizontal beam-hole sleeves in the beryllium were replaced by short sections, with a labyrinth seal between the beam tubes and the sleeves.

The present concept of the horizontal beam tube employs internal cooling, with coolant provided by primary water flowing axially through channels within the tube and discharging into a plenum connected to the vessel outlet line.

With the exception of the control-rod release mechanism, a prototype of the mechanical drive components is being procured for testing in the mockup. Detail drawings of the ball-latch release mechanism are now being completed.

Purchase contracts were placed for design and fabrication of the pressure vessel, primary circulating pumps, main heat exchangers, and pressurizer pumps.

Drawings were prepared and distributed which present the present mechanical and nuclear requirements for the fuel element. The drawings show the end adaptors proposed for the reactor.

Critical Experiments. – The completed fuel element for HFIR critical experiment No. 2 was flooded with water in the absence of beryllium reflector and found to have considerable source-neutron multiplication. It was made critical by removing part of the water from the central island. The fuel element has been placed in the equipment designed for this second series of critical experiments. Preliminary experiments to check out the controls and instrumentation and to establish initial critical conditions have begun. During this period of testing, the system was made critical with water in only the fuel region, and the control plates were set with a gray (nickel) window of 5.3 in., with the inner and outer black (copper-silver) sections inserted 6.7 and 8.0 in. respectively. The beryllium,

the control-plate region, and the target island were dry for this experiment.

MARITIME REACTORS PROGRAM

Physics. – One-dimensional radial burnup calculations with the CANDLE 2 code were performed for two Savannah-type cores. One core had the properties of a stainless steel egg-crate structure and had fuel elements of core I design containing uranium enriched to 4.2 wt % U^{235}. The other core had the properties of a Zircaloy egg-crate structure and contained the same fuel. Both cores appear to have about the same temperature reactivity deficit ($\Delta k \approx -0.06$), equilibrium xenon and samarium reactivity deficit ($\Delta k \approx -0.02$), and peak or override xenon reactivity deficit ($\Delta k \approx -0.01$). However, the Savannah core I control rods, which can shut down the stainless steel egg-crate core with a shutdown margin of about $0.02\Delta k$, can just barely hold down the Zircaloy egg-crate core. The burnup calculations indicate that the 4.2 wt % enriched-uranium fuel within a Zircaloy egg-crate structure would have about twice the reactivity lifetime that it would have within a stainless steel egg-crate structure. This increase in reactivity lifetime would result in about a 35% reduction in fuel-cycle costs for Savannah core I fuel. Reducing the initial enrichment of the Zircaloy egg-crate core to give about the same shutdown margin, with the central control rod stuck out of the core, as is now available in the stainless steel egg-crate core would result in an initial fuel enrichment of about 3.8 wt % U^{235}. This fuel loading in a Zircaloy egg-crate core would have a reactivity lifetime about 65% greater than that of a 4.2 wt % enriched fuel loading in a stainless steel egg-crate core and would result in about a 30% reduction in fuel-cycle costs.

ARMY PACKAGE POWER REACTORS

Three high-burnup fuel elements (18 Mwyr) from core I of the SM-1 reactor were inspected visually at the SM-1 site. The elements did not show any signs of damage. It is planned to place two of the elements back into the reactor with the core II loading of SM-1 for higher U^{235} burnup. These two elements will then be exposed to average U^{235} burnups beyond the burnup levels achieved in the two elements which are undergoing hot-cell examination at ORNL. Presently, information exists on ~20 and ~40 at. % average U^{235} depletion. The two high-burnup elements will be exposed to U^{235} depletions of ~60 and ~80 at. %.

The five Eu_2O_3–stainless steel dispersion control rods in the SM-1 were inspected after operating for approximately 8 Mwyr. The rods appeared to be in very good condition.

A spent fuel element (S-79) from core I of the SM-1 reactor is being examined in the ORNL hot cells. This is a conventional flat-plate unit fueled with fully enriched UO_2 dispersed in stainless steel and clad with type 304L stainless steel. The irradiation exposure was for 16.4 Mwyr (~42 at. % depletion of U^{235}). Inspections performed to date include determination of over-all bundle dimensions, rippling in top and bottom

plates, coolant-channel spacings, nature of radioactive deposits, and relative over-all burnups by gamma scanning. With the exception of rippling in the outermost plates of 23 mils maximum amplitude and radioactive deposits built up at the inlet end of the element, no unusual effects have been noted. The element has been disassembled into its component plates for further examination.

A drive shaft of type 17-4 PH stainless steel from the SM-1 control-rod drive system and the control-section basket made of type 304 stainless steel have been received at the hot cells and are being examined. The 17-4 PH component, originally age-hardened at 1000°F, and the basket have functioned successfully in a pressurized-water environment of neutral pH for 10 Mwyr. Detailed visual examination at 5 to 10× revealed no evidence of cracks or other forms of damage in either the basket or the shaft. Nondestructive surface examination with magnetic particles and dye penetrant remains to be performed on the drive shaft before it is sectioned for metallographic examination.

NUCLEAR TECHNOLOGY AND GENERAL SUPPORT

Reactor Evaluation Studies. — A study has been made of fuel fabrication costs as a function of fuel material and fuel-element design. A simplified approach was utilized which involved the separation of cost factors into two general groups: one containing factors independent of a specific reactor fuel assembly design (but limited to rod-in-bundle configurations), and the other containing design-dependent factors (primarily due to the method of assembly). Fuel elements for GCR-2, Yankee, Dresden, and CANDU reactor types were considered; fuel materials considered were slightly enriched uranium, natural uranium enriched with "commercially pure" plutonium, and natural uranium enriched with recycle plutonium. Cost equations were developed for the above cases; these include cost estimates for oxide powder production and fabrication, salvage recovery and recycle, fuel-element loading and assembly, quality control and inspection, and fuel losses.

Calculations were made for determining what effect increasing the fission cross section of Pu^{239} by 20% would have on the fuel value of plutonium. The reactor types studied were Dresden (H_2O) and CANDU (D_2O). It was found that changing the fission cross section increased the fuel value of fissile plutonium about \$2/g in the Dresden, but decreased the value about \$0.7/g in the CANDU (no fabrication-cost penalty was associated with recycle fuel). This was due to the relative influence of plutonium on the initial-cycle lifetime, in comparison with its influence in subsequent cycles. For the Dresden reactor, increasing the fission cross section of plutonium increased the recycle lifetime more than the initial-cycle lifetime, while the reverse was true for the CANDU reactor.

Radiation Detector Development. — Problems involving counting statistics and spectrometer response functions have long been recognized as difficulties in the path of complete understanding of experimental gamma-ray spectra. An equally important problem posed by the nature of the spectrometer output is under study at the Bulk Shielding Facility. Briefly, the problem involves the impossibility of exactly defining a continuous

spectrum by a finite number of discrete points, the form in which the spectrometer data are displayed by the usual multichannel analyzer. It has been found, assuming that counts and response functions are exactly known, that expressions approximating a single parameter dependent on the spectrum, such as dose, can be written as linear combinations of a finite number of response functions. The physically logical assumptions that $g(e) \geqq 0$ [$g(e)$ = particle spectrum as a function of energy] and $0 \leqq e \leqq \infty$ (nonnegativity) permit useful estimates of upper and lower bounds of the desired parameters. A method called "linear programming," taken from the mathematics of economics and scheduling, has been utilized in the computation of such bounds. The methods developed are expected to be useful in further study of the problems of counting statistics and response functions, and to aid directly in analysis of experimental results obtained with crude spectrometers such as threshold foils.

Chemical Behavior of Thermoelectric Materials. – In further studies of the characterization of nearly stoichiometric UO_2, estimates were made of the a_0 value of UO_2 unexposed to air and of the precision which could be achieved by using great care with the ordinary Debye-Scherrer powder diffraction method for determining a_0. Unit cell measurements were made on UO_2 which had been prepared by the hydrolysis of U^{4+} in a molten mixture of NaCl-KCl-UCl_4 and which had been protected from oxygen contamination during all subsequent handling procedures. In making the measurements, Cu_K radiation and a G-E powder camera were employed, corrections were made for film shrinkage, and extrapolation techniques were used in evaluating the data. A value of 5.472 ± 0.001 A was obtained; it corresponds closely to the previously reported value of 5.472 ± 0.002 A for $UO_{2.00\pm0.003}$ (exposed to air), obtained with only routine attention to the achievement of high precision.

Fuel Element Development. – An exploratory study was conducted to determine the effects of alloying elements in enhancing the elevated-temperature properties of thorium. With hot hardness and recrystallization temperature being used as criteria in preliminary tests, 45 alloys based on C, Al, Cr, U, In, Mo, Nb, Zr, Sn, Be, Ti, and V were examined. Hot hardness at 600°C, compared with that of unalloyed thorium, showed a fourfold increase when 4 wt % In was added and a threefold increase when 5 wt % Zr was added. At 750°C these improvements in hardness were even larger. A Th–0.2 wt % Be–0.2 wt % C alloy appears promising at 400°C, giving a 3-1/2-fold increase in hot hardness. The recrystallization studies indicated that in alloys containing indium, softening occurs at ~600°C, and in those containing zirconium at ~530°C. Limited hot tensile tests confirmed the strengthening effects.

Nondestructive Test Development. – A study of x-ray attenuation coefficients by scintillation spectrometry has been in progress on various alloys associated with power, compact, and research reactors. Materials examined have been types 304 and 347 stainless steel, Zircaloy-2, 1100 aluminum, and enriched (93% U^{235}) uranium–aluminum alloys containing 7.0, 18.6, and 24.8 wt % uranium.

The low-energy boundary conditions of 50 to 130 kev were based on the effective energy capacity possible from the available x-ray unit (Norelco 50 to 150 kvp). Background and scattered radiation were minimized

not only by the shielding of the x-ray tube and detector but also by narrow beam collimation from the x-ray source to the detector.

Detection equipment was primarily a NaI(Tl) crystal optically connected to a photomultiplier. The pulse from the phototube is fed through a preamplifier to a linear amplifier and pulse-height selector for the shaping and selection of pulses between two definite voltage levels. Output from this unit then goes to a linear count-rate meter and a Brown recorder.

For purposes of correlation, theoretical gamma-ray absorption coefficients have been calculated for each material. This absorption was calculated for each element that was present in the amount of 0.1 wt % or greater as determined by chemical analysis.

With the assistance of the Mathematics Panel, both the experimental x-ray attenuation coefficients and the theoretical gamma-ray absorption coefficients are being fitted to representative curves. The Oracle was programmed for six selected curves from each set of data to obtain by the method of least squares the best mathematical equation.

Fuel Cycle Technology. — Supercentrifugation of thoria sols prepared by the sol-gel process showed that the sol is optimum for addition of 3 to 5 at. % uranium, evaporation, and calcination to dense fragments suitable for vibratory compaction when it has an N/Th atomic ratio of 0.13 to 0.15 and the liquid phase contains $\sim 10^{-3}$ M nitrate and $< 10^{-4}$ M soluble thorium and has a pH of 3.1 to 3.8.

A third 1-kg lot of UO_2-ThO_2 containing 3.9 wt % of 93% enriched uranium was prepared by the sol-gel process for vibratory-compaction fabrication of irradiation test specimens. The N/Th ratio in the thoria sol was decreased from 0.19 to 0.15 by refluxing with formic acid. By the toluene immersion technique the particle density of the calcined fragments was 9.79 g/cc. Forty-five per cent of the particles were > 4 mesh, and 80% were > 10 mesh. Products were compacted by vibration to a bulk density of 8.4 or 8.5 g/cc in 5/16-in.-diam tubes.

Reactor Controls Development. — A new concept of a coincident and redundant safety system has been devised, in which the coincidence element is the magnet which supports the safety rod or actuates the scram latch mechanism. A prototype magnet has been built which incorporates a saturating magnetic shunt. Energizing one of its three identical coils will result in negligible lifting force on the rod because of the shunt. Energizing a second coil will saturate the shunt, permitting the magnet to exert rated force. Energizing the third coil will not substantially increase the force because of saturation in the magnet armature. The magnet is thus a two-out-of-three coincidence element, holding the rod if any two of its three coils are energized, and dropping the rod whenever any two coils are de-energized. The technique is applicable to magnets containing different numbers of coils.

The release time of the prototype magnet, tested with a load of 300 lb being held with a force of 600 lb, has been measured as 4.1 msec when all three coils are de-energized simultaneously, and as 8.1 msec when two of the coils are de-energized and the third one remains energized. The performance of the prototype coincidence magnet is thus quite satisfactory. These measurements were made by using the transistorized switching circuit described in ORNL-3048.

The advantage of this approach lies in the ability to apply a simple test signal to the input of a channel of safety instrumentation, confirming that as a result the corresponding coils in all the magnets are de-energized. The test can be made at any time without interrupting reactor operation, since de-energizing one coil of these magnets does not drop the rods. This ability to test the entire instrument channel while the reactor is in operation is most important in applying coincidence techniques to reactor safety systems. In other available coincidence systems, complicated and expensive pulsed testing equipment is necessary, and the tests encompass less than the entire instrument channel. The coincidence-type magnet reported here is thus fundamental to the "second generation" ORNL reactor safety system now under development.

Power Reactor Fuel Processing: Corrosion Studies. – In flowing modified Zirflex dissolver solutions Haynes 21 alloy showed no localized attack and a maximum corrosion rate of 0.67 mil/month in 490 hr; the rate for type 347 stainless steel was 5.87 mils/month in 304 hr. Corrosion rates of LCNA alloy, types 309SCb and 304L stainless steel, and Hastelloy F were all < 1.2 mils/month in both the solution and the vapor phase after ~30 hr exposure through six dissolution cycles.

In 90 vol % 21 N HNO_3–10 vol % 40 N H_2SO_4 at 50°C, INOR-8, LCNA, and CD4MCu were corroded at maximum rates of 0.11, 0.52, and 1.74 mils/month, respectively, in 672, 672, and 168 hr. The LCNA showed localized vapor-phase attack even though dry air was passed through the vapor space.

In dry HCl at 200°C (192 hr) Haynes 25, Nichrome V, INOR-8, and Pyroceram 9608 showed corrosion rates of < 0.01 mil/month. In 34-hr exposures at 600°C, rates for duplicate specimens (except Pyroceram) were 5.30 and 2.14, 5.63 and 1.52, 10.8 and 16.4, and 1.2 mils/month respectively. The weight loss of the Pyroceram is believed to have been caused by mechanical chipping of the edge of the specimen. In 24-hr exposures to dry Cl_2 at 200°C, nickel, Haynes 25, Nichrome V, and INOR-8 gained weight, but Pyroceram showed no change. At 350°C all weight losses were equivalent to corrosion rates of ≦0.08 mil/month, and at 600°C (duplicate specimens except for Pyroceram), rates were (mils/month): 1.08 and 11.37 for nickel, 0.60 and 35.2 for Haynes 25, 0.38 and 8.79 for Nichrome V, 2.31 and 27.14 for INOR-8, and 0 for Pyroceram. In all cases attack appeared uniform. Rates were maximum for downstream specimens.

In boiling 4 M HNO_3 welded INOR-8 showed excessive rates (> 200 mils/month) in a 1.5-hr exposure. Unwelded Nichrome V showed uniform rates of ≦0.22 mil/month in a 336-hr exposure.

Analysis of erratic corrosion performance of titanium in UO_2 core dissolvent solution (35°C tests for multipurpose PRFP centrifuge) showed the effects to be due to small differences in solution composition affecting the concentration of the free fluoride contaminant. In tests with no fluoride the maximum rate was 0.03 mil/month; in some solutions rates were as high as 8.9 mils/month; complexing the F^- with Al^{3+} or Fe^{3+} decreased the rates.

Power Reactor Fuel Processing: Zirflex Process. – In an engineering-scale test of the modified Zirflex flowsheet with unirradiated TRIGA fuel, aluminum-clad 8% U–92% ZrH, the aluminum cladding and the Al-Sm neutron-poison disks dissolved in < 30 min in refluxing 2 M NaOH–1.8 M $NaNO_3$, and 65% of the two graphite plugs disintegrated in 2 hr in 90 vol % HNO_3–10

vol % H_2SO_4 at room temperature. The U-ZrH was 99.75% dissolved after 11.5 hr of refluxing in dissolvent initially 6.3 M NH_4F–0.55 M NH_4NO_3–0.01 M H_2O_2. Both acid-aluminum-stabilized solvent-extraction feed solution containing 0.46 M Zr, 0.78 M free F^-, 3.44 g of U per liter, and 0.8 M HNO_3 and $Al(NO_3)_3$, and H_2O-stabilized solutions containing 0.444 M Zr, 0.62 M free F^-, and 3.54 g of U per liter remained stable 300 hr at room temperature. When NH_4NO_3 was omitted from the dissolvent, 8% U–92% Zr plate dissolved in 2 hr, with no stable foam, although 2 moles of H_2 was evolved per mole of Zr dissolved. The scrubbed off-gas contained 99% H_2 and H_2O vapor and < 0.15% O_2.

The addition of ammonium tartrate to the dissolvent resulted in a more uniform dissolution rate, a lower final F/Zr mole ratio, and a higher zirconium concentration. The final solutions were stable at pH 5 to 10. The initial dissolution rates of Zircaloy-2 in 6 M NH_4F–0.35 M ammonium tartrate and in 6 M NH_4F were both 16 mg cm^{-2} min^{-1}, dropping to 8 mg cm^{-2} min^{-1} when the F/Zr ratio had decreased to 50 and finally to 0.6 mg cm^{-2} min^{-1}. The final F/Zr ratio was 4.0 (1.2 M Zr) when the dissolvent was 0.75 M in ammonium tartrate; it was 7.0 for NH_4F alone.

A solution of 0.7 M Zr, 5.2 M F, and 0.35 M ammonium tartrate at 30°C was exposed to a Co^{60} gamma radiation field of ~1 w/liter (about 100 times the radiant energy absorption expected in Zirflex waste) for two weeks with no apparent instability or gas evolution. A sintered UO_2 pellet exposed to a sample of the same solution at 98°C lost no weight in 67 hr.

Preparation of stable basic solutions from modified Zirflex waste required a pH of > 10 to ensure aluminum solubility, and the soluble zirconium tartrate complex decomposed to insoluble zirconium oxide. The logarithm of the time required for this decomposition was a linear function of the tartrate/Zr ratio. Extrapolation of the data indicates that a solution containing 2 moles of tartrate per mole of Zr at pH 10 would be stable for two years.

Power Reactor Fuel Processing: Solvent Extraction. – In continuing studies on niobium extraction chemistry, the extraction coefficient for niobium from 2 M HNO_3 into 30% TBP–dodecane, in which 10% of the TBP was degraded by refluxing 6 hr with 6 M HNO_3, was 0.26 when all the niobium was in an extractable form. The coefficient in organic-to-aqueous experiments was 0.33.

The use of an acid-deficient, instead of an acid, feed with a Purex type of flowsheet improved decontamination from ruthenium and zirconium-niobium by factors of about 10 and 4, respectively, with no increase in uranium loss. This improved uranium solvent-extraction method may be used to recover uranium, but it is not applicable in its present form to plutonium extraction. The scrub was 2 M HNO_3. The feed in the acid experiments was 1 M HNO_3 and in the acid-deficient experiments was 0.18 N acid deficient, with additional acid added to the fourth extraction stage.

In the intermediate-scale mixer-settlers operating with the standard Purex flowsheet, stage efficiencies were up to 93% for uranium extraction and up to 95% for uranium stripping with modified impellers operated at speeds up to 1200 rpm. The extraction-scrub bank was operated at an aqueous throughput of 92 ml/min (85% of flooding capacity), the back extraction-partitioning bank at an aqueous throughput of 25 ml/min (~23% of flooding capacity), and the stripping bank at an aqueous throughput of 350

ml/min (~90% of flooding capacity). The uranium loss in extraction was 0.001% in five stages, and in stripping was 0.001% in seven stages. Operation was stable at an aqueous feed throughput of 52 ml/min, equivalent to a uranium throughput of 23.7 kg/day. Aqueous throughput in the stripping bank was limiting at 350 ml/min (90% of flooding capacity).

Power Reactor Fuel Processing: Uranium Carbide and Uranium-Graphite Fuels. – When uranium-graphite fuel specimens irradiated to ~0.001% burnup were burned in a stream of oxygen at 900°C, the amount of Ru^{106} volatilized appeared to be a function of the uranium content of the fuel. With specimens containing 3 and 11% uranium, 60 to 95% and 30 to 40%, respectively, of the Ru^{106} were volatilized.

More than 99% of the uranium was solubilized from a Pebble Bed Reactor fuel specimen containing 125-μ UO_2 particles coated with 17-mil-thick Al_2O_3 when the specimen was ground to < 200 mesh and leached 6 hr with boiling 15.8 M HNO_3.

Uranium loss to the graphite residue was only 0.012% from a TURRET fuel specimen (graphite impregnated with uranyl nitrate solution) in the standard two-leach treatment with boiling 90% HNO_3.

The reaction of uranium monocarbide with water at 60°C yielded virtually the same reaction products as at 80°C. The only temperature effect was a marked decrease in reaction rate at the lower temperature: a 5-g specimen reacted completely in 2 hr at 80°C, but only 95% in 6 hr at 60°C. At 60°C the average off-gas volume in four experiments was 92.8 ml (STP) per gram of specimen; its composition was 11.1 vol % hydrogen, 86.2% methane, 1.8% ethane, 0.54% propane, 0.28% butane, 0.071% pentanes, 0.024% hexanes, and 0.02% of an unidentified material. At 80°C the off-gas volume was 93.0 ml (STP) per gram of specimen; its composition was 10.4% hydrogen, 86.7% methane, 1.8% ethane, 0.69% propane, 0.27% butane, 0.059% pentanes, and 0.055% hexanes. There was no evidence of a change in reaction products with temperature.

Power Reactor Fuel Processing: Zircex Process. – The $ZrCl_4$ waste from the Zircex process may be made less corrosive by removing the chloride with H_2O_2. The contacting of 1.33 M $ZrOCl_2$–8 M HCl with 10 M H_2O_2 was rapid and stoichiometric at 75 to 99°C according to the equation $2HCl + H_2O_2 \rightarrow Cl_2(gas) + 2H_2O$ until the total chloride concentration, based on the original volume, was decreased to 1.1–1.5 M. Further chloride removal was hindered by the increasing viscosity of the solution and finally the formation of a hydrated solid, probably zirconium peroxide, which dissolved readily in nitric acid. When enough nitric acid was added with 100% excess hydrogen peroxide to make the product 0.6 M in HNO_3, the chloride concentration was further decreased to 0.5 M. After chloride removal, stable basic solutions were made by adding KOH-K_2CO_3-tartrate solution. The same chloride removal reaction proceeded in the presence of nitric acid except when dissolved iron was present, in which case the major reaction was decomposition of hydrogen peroxide to water and oxygen.

Power Reactor Fuel Processing: Mechanical. – Eighteen unirradiated NaK-bonded SRE fuel rods were satisfactorily processed in final cold testing of the dejacketing equipment. Approval to operate with irradiated fuel was granted after satisfactory demonstration of the CO_2 fire-extinguishing and double-containment system.

In a four-stage model-leacher dissolution run, in which nine batches of UO_2 pellets, 600 g per batch, were dissolved in 4 M HNO_3 at an HNO_3/UO_2 mole ratio of 5.43, dissolution was 98.4 to 99.3% in 4 hr. In two runs with 8 M HNO_3 the steady-state composite product contained 500 g of U per liter and 1.9 M H^+ at an HNO_3/UO_2 mole ratio of 4, as compared with a composite product of 335 g of U per liter and 4.1 M H^+ at an HNO_3/UO_2 mole ratio of 6.15. In each run apparent steady state was attained in three to four stages.

Power Reactor Fuel Processing: Beryllium-Containing Fuels. – In boiling nitric acid–sulfuric acid mixtures, 70% BeO–30% UO_2 GCRE fuel pellets dissolved completely in < 23 hr. Dissolution rates were essentially the same in 6 M HNO_3 containing 0.5 to 2 M H_2SO_4 and in 8 M HNO_3–0.5 M H_2SO_4. In the presence of sulfuric acid, BeO dissolution rates were higher than in 8 M HNO_3 alone. In all cases uranium was leached completely from the beryllia matrix in 5 to 7 hr.

The dissolution rate of high-fired beryllia was less than 0.01 mg cm^{-2} min^{-1} in boiling 30% sulfuric acid, 2.2 M ammonium fluosilicate, dimethyl sulfoxide, and resorcinol.

Fluoride Volatility Processing. – In a total of seven complete flowsheet runs made to date with nonirradiated enriched-uranium–zirconium alloy fuel elements, nonrecoverable uranium losses ranged from 0.18 to 1.94 g (0.04 to 0.31%) and averaged 0.75 g. Total cation impurities in the UF_6 product were less than 320 ppm. A system is being constructed for continuous flushing of the off-gas line with liquid HF to eliminate solids from the off-gas.

PHYSICAL RESEARCH PROGRAM

Reactor Operations. — Better performance has been obtained from the revised ball-latch mechanism in the ORR control rods. In spite of considerable trouble with the mechanisms and two bimonthly shutdowns in the last quarter, an operating time of 80% was achieved at the ORR.

Work is continuing on the proposal to increase the ORR power to 40 Mw.

Waste Disposal. — A good balance was obtained between the different sources of strontium entering White Oak Creek and that flowing across the White Oak Creek Dam. In the case of ruthenium, only about half of that identified as entering the system was accounted for in the dam discharge.

Considerable waste is seeping from open waste pit No. 4, and this accounts for most of the ruthenium entering the creek. Practically no strontium is leaving the pits, however.

Hot-Cell Operations. — The load is increasing in the Physical Examination Hot Cells, Buildings 3025 and 3026D. Some overtime work has been necessary, and as soon as personnel are available it is planned to begin a second shift.

Plate-spacing and gamma-scanning measurements of SM-1 fuel elements are almost complete.

The mechanical disassembly of SRE fuel elements in cell A of Building 3026D is almost ready for application to radioactive fuel. Cold runs have been in progress, and several mechanical troubles are being corrected.

PHYSICS AND MATHEMATICS RESEARCH

Fast-Chopper Time-of-Flight Spectrometer. — Additional neutron radiative capture measurements were made with the 28-in.-diam spherical liquid scintillation detector [Bull. Am. Phys. Soc. 4, 474 (1959)] installed at the 11.5-m flight path of the ORNL fast chopper. The radiative capture cross sections of ~25 naturally abundant elements in the atomic-weight range $90 < A < 200$ were measured from ~200 to 9000 ev with a resolution of 0.17 μsec/m. These cross sections agreed to within 20 to 25% with the reported Van de Graaff results [Phys. Rev. 122, 182 (1961)] in the 9000-ev region.

The 28-in.-diam scintillator was recently moved to a 25-m flight station at the General Atomic linear accelerator for testing the feasibility of using this type of equipment in the presence of the intense "beam flash" produced by electron linear accelerators. Preliminary tests with a 20-Mev electron beam (5-μsec-wide electron pulse, 70-ma electron current in the pulse) indicated that this scintillation equipment recovers quite rapidly (≤ 8 μsec) after the beam flash and is well suited for measurements with these accelerators.

High-Voltage Experimental Program: Van de Graaff Conversion. — The 3-Mv pulsed Van de Graaff has been shut down for major changes. The mass-analyzing magnet is being remounted on a Navy surplus gun mount and thus will be able to be rotated, obviating the need for a "fanning" magnet.

The vacuum system is being replaced with higher speed diffusion pumps with lower back-streaming characteristics. The high-voltage terminal is being converted from an rf source to a duoplasmatron ion source. The new system should afford several times more peak ion current at pulse widths less than 5 nanoseconds.

High-Voltage Experimental Program: Neutrons from the He^3 Bombardment of Some Light Nuclei. — The angular distributions of neutrons from $C^{13}(He^3,n)O^{15}$ and $Be^9(He^3,n)C^{11}$ were measured at a bombarding energy of 2.66 Mev. The excitation function of the $C^{13}(He^3,n)O^{15}$ reaction was measured at 90° for He^3 bombarding energies from 1.0 to 2.66 Mev. The neutron spectra for the $C^{13}(He^3,n)O^{15}$ reaction showed the ground state and the third excited state (6.15 Mev) to be clearly resolved at a flight path of 2 m. The angular distribution for this reaction (ground-state neutrons) showed a pronounced peaking at $\theta_{c.m.} = 0°$ and 100° and a minimum at $\theta_{c.m.} = 45°$. For the reaction $Be^9(He^3,n)C^{11}$ the ground state and the first fine excited states were observed. Data for the above reactions are now being fitted to distorted-wave calculations from IBM 7090 share code Sally.

Spectroscopy Research Laboratory. — The triatomic metaborate ion, BO_2^-, was isolated in solid solutions in NaCl, NaBr, KCl, and KBr lattices. The infrared spectra of these solutions were studied, and the ion was shown to be linear and to closely resemble CO_2. Fundamental frequencies identified with ions containing B^{10}, B^{11}, O^{17}, and O^{18} were measured, plus several overtone and combination bands of the more abundant species. For $(B^{10}O_2^{16})^-$ and $(B^{11}O_2^{16})^-$ in KCl, observed frequencies are $\nu_1 = 1072\ cm^{-1}$, $\nu_2 = 610$ and $590\ cm^{-1}$, $\nu_3 = 2043$ and $1972\ cm^{-1}$.

CHEMISTRY RESEARCH

Solvent Extraction Research. — Uranium was recovered from modified Zirflex dissolver solution (no nitrate) by synergistic Dapex extraction. From nearly saturated solution (2.7 M F, 1.5 M NH_4, 0.33 M Zr, 0.004 M U), uranium extraction coefficients were ~2 with either 0.1 M di(2-ethylhexyl)phosphoric acid (D2EHPA)—0.05 M tri-n-octylphosphine oxide (TOPO) or 0.3 M D2EHPA—0.15 M diamyl amylphosphonate (DAAP). The coefficients increased by more than an order of magnitude on 1:1 dilution of the Zirflex solution with water. The extracted uranium was readily stripped with ammonium carbonate solution. Zirconium extraction was low, $E_a^o < 0.15$, from the undiluted solution. TOPO alone also extracted uranium but only in the presence of some nitrate; for example, $E_a^o \approx 2$ with 0.3 M TOPO when 0.3 M HNO_3 or $NaNO_3$ was added to the Zirflex solution.

In development of flowsheets for final-cycle plutonium recovery by extraction with amines, the effects of extractant degradation on the ability to reject fission products are being examined by measuring the extraction of Zr^{95}-Nb^{95} tracer from nitric acid solution. Prolonged contact with nitric acid gives measurable Zr-Nb extractability, which increases with time and with nitric acid concentration, and apparently

is due more to changes in the amine (trilaurylamine) than in the diluent (Amsco 125-82 plus tridecanol). Several scrubbing agents (e.g., sodium hydroxide, acidic sodium fluoride) removed the Zr-Nb without decreasing subsequent extractability. Treatment with dilute sodium chromate (0.005 to 0.1 M) in basic solution decreased the subsequent Zr-Nb extractability; this solution appeared promising for process cleanup.

Ion Exchange Studies. — Calculations of predicted loading rates for uranyl sulfate on chloride-equilibrated 960-μ Dowex 21K were completed. Assuming that the kinetic process involves diffusion of the $UO_2(SO_4)_2^{2-}$, SO_4^{2-}, and Cl^- ions, the loading rate was shown to be a function of the ratio of equilibrium sulfate to uranyl disulfate. Experimental loading data could be fitted by the proposed model when this ratio was 0.02. Adequate experimental methods of determining resin bisulfate loadings are not yet available, but the value 0.02 is within the range which available equilibrium data require, 0.00 to 0.25, and it allows reasonably accurate predictions of the rate at which chloride ions are eluted from the resin during uranyl sulfate loading. The model was further confirmed by the absence of significant variation in experimental loading rates for uranyl sulfate on chloride-equilibrated resin over the concentration ranges 0.003 to 0.006 M uranyl sulfate and 0.025 to 0.200 M total sulfate.

Analyses of Dowex 50W-X8 (20 to 50 mesh) resin which had been placed in a circulating system of demineralized water and irradiated in a 10,000-curie Co^{60} source for a dose of 1.1×10^9 r showed a 30% decrease in specific resin capacity for a 20% decrease in resin volume, and a decrease in the moisture content from its original 42.7% to 38.6%. A dose of 5.1×10^9 r decreased the specific capacity 60% and the resin volume 85%, but increased the moisture content to 55.1%.

Mechanisms of Separations Processes. — In studies on separation by foam, measured surface tensions over the experimental range 40 to 55 dynes/cm agreed with values calculated from the Gibbs-Langmuir equations. Aqueous solutions of each of four surface-active alcohols — n-butanol, n-pentanol, n-hexanol, and n-heptanol — and 30 solutions containing three and four of these alcohols were tested.

Chemical Engineering Research. — A pulsed column being operated at low aqueous/organic flow ratios typical of the scrub section was studied to determine the effect of type of plate and dispersion on stage height. The HETS (height equivalent to a theoretical stage) values for stripping nitric acid from 30% tributyl phosphate in kerosene in a nozzle-plate column being operated with the solvent phase continuous were 1.6 ft at an aqueous/organic ratio of 1/7 and 2.9 ft at 1/20. These values were slightly greater than those for a sieve-plate column with the aqueous phase continuous. The HETS for the sieve-plate column being operated with the solvent phase continuous was much greater, 4.8 ft at an aqueous/organic ratio of 1/7.

Physical Chemistry of Molten Salts. — In the program of cryoscopic measurements of fluoride systems, the effects of other alkali-metal fluorides on the freezing point of NaF were investigated. When the concentrations of any of the alkali fluorides were 6 mole % or less, the measured freezing points were equal to those expected of ideal solutions. For LiF and KF solutes the ideal behavior appeared to extend up to 15 mole %.

When deviations from ideality occurred, they were negative for LiF and positive for KF, RbF, and CsF. At the same concentration in the non-ideal range, the magnitudes of the positive deviations followed the order CsF > RbF > KF.

As was the case with the tetravalent fluoride solutes previously reported (ORNL-3048), the cation size appears to be important. When the ionic radius of the solute metal was smaller than that of Na^+, negative deviations occurred, and vice versa, with a correlation between the difference in ionic radii and the deviations from ideality. All these binary alkali-fluoride mixtures containing more than 40 mole % NaF yield pure crystalline NaF as the primary phase on cooling from the liquid state, permitting the evaluation of partial molal free energies of mixing with NaF over wide ranges of composition. These free energies should be useful for ultimate correlation with structural parameters of the solutes.

High-Temperature Aqueous Solution Chemistry. — Studies of the chemistry of protactinium in aqueous systems at elevated temperature were initiated on a macro scale with the use of Pa^{231} recently made available from British sources. Preliminary experiments demonstrated that, at concentrations of ~100 ppm, protactinium was not precipitated rapidly from (i.e., was apparently soluble in) 2 M $Th(NO_3)_4$–3 M HNO_3 or 2.5 M $Th(NO_3)_4$–0.5 M HNO_3 solutions at temperatures from 21 to 180°C. These tests were performed with stainless steel containers and, in one case, in the presence of zirconium metal and zirconium oxide. These preliminary results suggest that the adsorption of protactinium on these metals and corrosion products is either slow or negligible in solutions of the type tested.

Nuclear Methods of Chemical Analysis. — Radioactivation analysis methods, involving the use of short-lived radionuclides having half-lives in the range 0.89 sec to 37 min and direct radioactivity measurement techniques by means of a gamma scintillation spectrometer, have made it possible to determine many different elements in a large variety of sample materials. Analyses for the elements Li, W, N, O, F, S, In, Hf, Se, Sc, Ag, Rh, Rb, Dy, Ir, V, Al, Hg, Nb, Mg, Co, Ta, Re, Br, Ga, Sb, Sm, U, Th, Pt, Cl, Pd, and Zn are now being made by means of short-lived radionuclides and by the nondestructive assay of the irradiated sample material. Metals, alloys, animal tissue, body fluids, feed materials, ores and minerals, and chemical compounds are now being speedily analyzed in an almost routine manner. The fast pneumatic tube system of the HN-3 laboratory at the ORR is being used in this work. The system has an average flux of 5×10^{13} thermal neutrons cm^{-2} sec^{-1}, and it has been possible, depending upon the irradiation time, decay of the radionuclide after irradiation, and the efficiency of the counter, to determine microgram and submicrogram amounts of the elements listed. For example, short-lived radionuclides can be used to determine quantitatively 10^{-10} g of Nb, 10^{-13} g of V, 10^{-14} g of In, 10^{-8} g of Se, 10^{-10} g of W, 10^{-5} g of S, and 10^{-4} g of O.

Chemistry of the Heavy Elements. — The reduction and the reoxidation of an aqueous solution composed of UO_2SO_4 (0.0123 m), $CuSO_4$ (0.0085 m), and D_2SO_4 (0.0072 m) in D_2O (98.5 at. % D) are being studied in the wavelength range 0.34 to 1.2 μ as a function of temperature, time, and overpressure of H_2 and/or O_2. The second prototype high-temperature aqueous spectrophotometric cell, designed to allow gas-liquid equilibration, is being used.

At 25°C and 750 psi H_2 the Cu(II) and U(VI) were found to be reduced successively to Cu(I) or Cu(0) (during the first 19 hr) and U(IV) (during the next 28 hr). The disappearance of the copper spectrum occurred without detectable turbidity, and it is presumed that the reduction of copper occurred at the surface of the titanium cell wall. The U(IV) species (unhydrolyzed and hydrolyzed) appeared to be essentially soluble at room temperature, and their spectra were qualitatively identified and followed subsequently as a function of temperature over the range 4 to 130°C. The spectral data indicated that the UOH^{3+}/U^{4+} ratio increases montonically with temperature, while the total U(IV) in the reduced solution slowly decreases due to hydrolytic precipitation.

Aggregation of the U(IV) hydrolytic species appeared to occur preferentially at the surface of the cell walls, because the changing spectrum of the system was uncomplicated by turbidity when the temperature was raised slowly (i.e., < 0.1°/min). At higher rates of heating, turbidity did occur; for example, at 37°C, after a 1°/min rise from 25°, the absorbance at 7150 A increased from ~0 to > 2.4 in 2 min. At 130°C not more than 15% of the U(IV) remained in solution. This hydrolysis was found to be completely, though slowly, reversible with temperature.

Reoxidation of the system at 25°C and 180 psi O_2 was rapid. The conversion, U(IV) → U(VI), was 96% completed within 5 to 6 min. Oxidation of the copper proceeded more slowly, approximately 3-3/4 hr being required for completion.

Reduction of the solution with H_2 at 150°C and higher resulted in complete elimination of all species absorbing in the wavelength range studied (viz., 0.34 to 1.2 μ). Upon reoxidation at high temperature, unidentified absorbing species were observed which could be copper and/or uranium intermediates or corrosion products.

Electrochemical Kinetics and Its Application to Corrosion. — An oscilloscopic technique was developed and used in a study of the differential capacity of electrodeposited iron electrodes actively corroding in halide solutions. Significant differences in capacity are found which depend primarily on the identity of the anion contained in solution, and depend only slightly on acidity. Differential capacities in acid halide solutions decrease in the order HCl > HBr > HI; likewise, spontaneous corrosion rates decrease in the same order, iodide being an effective corrosion inhibitor. In addition, capacities increase with time, and the rate of increase is proportional to the corrosion rate. The change of capacity with time seems to depend on the amount of hydrogen produced by reaction at the interface and absorbed by the metal, as well as on the surface roughness, which increases with time due to corrosion. In support of the electrochemical measurements, electron microscopic examination of electrodes reveals differences in surface topography which are characteristic of the anion in solution.

Studies were continued on the role of the anions OH^-, SCN^-, Cl^-, and SO_4^{2-} in affecting the reduction kinetics of cupric ion on passive stainless steel in aqueous perchlorate media. Potentiostatic measurements demonstrate that adsorption of anions from solution onto the surface of passive stainless steel follows, to a first approximation, a potential-dependent Langmuir isotherm. At constant potential, the ease of adsorption of the anions studied decreases in the order $OH^- > SCN^- > Cl^- > SO_4^{2-}$. The

apparent specific rate constant for reduction of cupric ion increases with increasing anion adsorption, particularly OH^-; and the slopes of potential plotted vs logarithm of current density change with extent of adsorption, in harmony with a change of mechanism with changing surface constitution. The present technique permits a determination of energetic quantities pertaining to the adsorption reaction, and these were determined for the adsorption of OH^- on passive stainless steel in solutions of interest.

Chemical Applications of Nuclear Explosions. — Measurements of tritium exchange when an H_2-H_2O-HTO mixture is passed over layers of $CaSO_4$ showed that the process is diffusion-controlled. The maximum exchange observed under these conditions was 25% for 45 sec residence time at 600°C with flow rates of 6.5×10^{-3}, 4.5×10^{-4}, and 4.1×10^{-11} mole/min of H_2, H_2O, and HTO respectively. The calculated equilibrium exchange under these conditions is 90%. The exchange was much more rapid with forced gas flow through a fixed bed of $CaSO_4$, 48% at 600°C in 5 sec compared with 16% in identical experiments in which the gas was passed over layers of $CaSO_4$. The results indicate that if mixing is good in the Gnome experiment, extensive exchange of tritium with environmental water will occur.

METALLURGY AND MATERIALS RESEARCH

Ceramics Research. — The sinterability of similarly pressed compacts prepared from thoria powders with different properties was determined as a function of particle size, crystallite size, surface area, immersion density, and particle size after treatment for 3 min in a Waring Blendor. The sinterability, as indicated by density after firing at 1800°C for 2 hr, decreased gradually with increasing particle size, but scatter of points far exceeded experimental error. For surface area, crystallite size, and immersion density, there was a rough correlation with sinterability for powders in which the precipitation technique varied. However, for sinterability of powders with properties changed by higher calcination temperature, there was an abrupt departure from the correlation. The best correlation of sinterability with powder properties was with particle size after Waring Blendor treatment, which represents a measure of particle integrity. The larger the resultant particle size after blending, the lower the sinterability of the powder from which the sample was taken.

Structure of Metals. — Studies of annealing under an applied stress showed two effects. First, recrystallization in high-purity aluminum was appreciably retarded at 240°C by the application of a compressive stress. In addition, the fraction of recrystallized grains that had a $< 100 >$ fiber texture was greater in the specimens annealed under stress.

CONTROLLED THERMONUCLEAR RESEARCH

DCX-1 Facility. — Fabrication of the electric quadrupole lens was completed, and the lens was installed at the base of the DCX accelerator tube. The ion source and accelerator tube assembly was completely realigned to correct for the effects of the fringing magnetic field on the

H_2^+ beam trajectory. As a result of the additional focus control and the realignment, 15-ma H_2^+ has been injected into the plasma region, although not as yet with entirely acceptable focus. The addition of a $1 \times 1/2$ in. aperture at the entrance to the plasma region renders the focus acceptable and still allows the injection of 10-ma H_2^+.

Some preliminary runs have been made for measuring plasma rf radiation at the proton cyclotron fundamental and several harmonics as a function of pressure with gas dissociation. It was found that the intensity of the radiation at each frequency varied nonlinearly with the pressure.

Preliminary work has also been done on probing the plasma potential with a variable-energy lithium ion beam and with measurements of slow ions drifting out of the plasma region. The measurements to date, all at 2×10^{-6} mm Hg, indicate a potential of about 100 v at 2 ma of injected H_2^+ current. The potential increases with injected current to a value of about 300 to 400 v at 8 ma of current.

DCX-EP-A. — The electron gun (which is part of the 100-kev neutralized accelerator) has been installed in the EP-A facility, but it has not as yet been run because of incomplete data for the magnet coils. Magnet coil tests are in progress and are expected to be completed by the first of June; tests on the electron gun, the 100-kev grid source, and the 100-kev neutralized beam source will then be run.

Work has been resumed on the installation of the required isolation transformer operated by the 100-kev neutralized beam source.

Data from the Sherwood test facility indicate good vacuum pumping by the gettering action of the titanium rotating anode.

During the previous month, the high-voltage power supplies for the Sherwood test facility were increased in drain potential by a factor of 2, which will enable further testing of the low-energy prototype of the 100-kev neutralized ion beam accelerator.

High-Current Injection. — The 600-kv beam facility has produced a beam of 115 ma at 600 kev. Profile measurements have been made along an 8-ft path of the beam by using probes. It appears to be completely space-charge-neutralized in the region below the lens, except that the H^+ component is dispersed below the focal point when it is focused above the target. There have been no target failures since modification of the facility.

It has been demonstrated in the 100-kv test facility that automatic space-charge neutralization occurs in an ion beam which has been passed through a solenoidal magnet field, if no electric fields are present to drain electrons from the beam. A positive bias on the target, for example, destroys the neutralization. The beam remains neutralized, however, between a region of transverse magnetic field introduced above the target and the solenoid even with the target biased.

Theory. — A solution of the statistical equations for plasma and radiation has yielded a formula for cyclotron radiation which differs from earlier ones by Trubmikoff and by Rosenbluth and Drummond. The origin of this difference is not yet known.

The cross section for the dissociation and ionization of the H_2^+ molecule by electrons has been calculated by the Gryzinski formalism. The results differed considerably from those obtained by the Born approximation.

A sufficient criterion for electrostatic stability of plasmas has been found which requires that the operator appearing in the linearized Volsov equation have an anti-Hermetian representation. With this criterion, stability has been proven for a class of equilibrium distributions isotropic in velocity but inhomogeneous in space.

BIOLOGY AND MEDICINE PROGRAM

RADIATION EFFECTS ON BIOLOGICAL SYSTEMS

Pathology and Physiology. — In order to study the influence of dose rate on the oncogenic effectiveness of ionizing radiation, RF male and female mice were exposed to whole-body Co^{60} gamma radiation at varying dose rates, beginning at 10 weeks of age. The dose rate ranged from less than 1 rad/day (administered continuously 23 hr daily) to 7 rads/min. Mice were exposed daily until they had accumulated a predetermined total dose or until half the starting population had died. In general, the life-shortening and oncogenic effectiveness of the radiation decreased with decreasing dose rate, but the precise relationship between effectiveness and dose rate varied from one effect to another. For induction of thymic lymphomas, intermediate dose rates of 20 to 75 rads/day were maximally effective. All the effects studied were observed to be induced at a rate of 5 rads/day, but none were found to be induced at a rate of 1 rad/day, even when the mice were exposed throughout their entire life span. For statistical and other reasons, however, the significance of the negative findings is equivocal.

Biophysics: Magnetic Resonance Studies. — A preliminary experiment on the production of electron spin resonances in glycine by energetic protons from the ORNL 86-in. cyclotron was performed. Doses, approximated from beam current measurements with a Faraday cup and calculation of stopping powers for protons of known energy, were from 3 to 50 megarads delivered at about 1-1/2 to 2-1/2 megarads/min. By the use of aluminum attenuation, two linear energy transfer (LET) distributions (19- and 10.5-Mev incident protons) were employed with LET averages of roughly 4.6 and 13 kev/μ. More thorough dosimetric studies are in progress. The electron spin resonance (ESR) dose curve and subsequent decay are quite similar to results obtained with 300-kvp x rays, for which the average LET is very roughly 3.5 kev/μ. Specifically, there is saturation at doses above 20 megarads and slightly more rapid decay (on semilogarithmic plots) of high-dose samples than of low-dose samples, as was seen in previous x-ray experiments. No marked differences have been noted between the results of the 10- and 19-Mev protons.

Free radicals produced in succinic acid and di-succinic peroxide were studied by ESR techniques. While the ESR signals for these two materials differed in shape, the efficiency for their production was about the same at high x-ray doses. As decay proceeded, succinic acid showed a marked increase in fine structure, but the peroxide showed no change in curve shape.

Biophysics: Ultraviolet Studies. — Deoxycytidine was irradiated by monochromatic ultraviolet light obtained by use of the large quartz monochromator. The destruction of the pyrimidine ring was detected by the change in its absorption spectrum. For incident wavelengths of 2800, 2650, 2480, 2380, and 2300 A, the quantum yield for destruction is $(6.0 \pm 0.1) \times 10^{-3}$. At 2250 A the yield is 7.1×10^{-3}. Irradiations at the longer wavelengths yield products which revert, on heating, to deoxycytidine. Irradiation at 2250 A gives rise to products which are not heat-reversible.

Work on the synergism observed in the combined action of x rays and ultraviolet radiation on *Tradescantia* pollen was continued, with emphasis on a determination of the action spectrum for the phenomenon. Preliminary results indicate that ultraviolet wavelengths in the range 2480 to 3650 A are effective in producing the synergism. The action spectrum deduced from these incomplete exploratory measurements appears to be a very broad curve covering the entire 2400- to 3650-A region.

Chemical Protection and Enzyme Catalysis. — The reversal of the pharmacological effects of S,2-aminoethylisothiourea (AET) by drugs has been studied at several levels of AET administration; studies were also made when the AET salt and the rearranged mercaptoethylguanidine were administered. Although nearly complete reversal of the oxygen tension effects of AET was obtained with the use of a vasodilator, no differences in protective action were observed.

A glycoprotein possessing thyroxine binding activity has been isolated from bovine plasma and purified by column chromatography. Its physical and chemical properties are currently being investigated.

Continuing structural studies on bovine heart malic dehydrogenase (mitochondrial) have shown the N-terminal amino acid to be glycine. Carboxymethylated enzyme was hydrolyzed, and 1.3 residues of S-carboxymethylcysteine were isolated per mole of malic dehydrogenase. This finding is compatible with the hypothesis that the coenzyme binds primarily to the free sulfhydryl groups of the enzyme.

COMBATING RADIATION DETRIMENTAL EFFECTS

Mammalian Radiation Recovery. — Cytotoxic antibody techniques have been used to identify lymphopoietic, granulopoietic, and erythropoietic elements in bone marrow, lymph node, and spleen suspensions of parent strain mice that were lethally irradiated and were treated with 20 or 40 million leukocytes from the peripheral blood of normal F_1 hybrid mice.

Rat urine contains substances that immunize mice and prevent the transplantation of rat bone marrow in the mice after lethal irradiation. Attempts are being made to identify the material chemically.

Studies of x-ray-induced chromosome aberration show that peripheral leukocytes present in freshly drawn human blood are all in the pre-DNA (deoxyribonucleic acid) G-I phase. Studies are in progress with H_3 thymidine to determine when DNA synthesis occurs and also whether the cells can divide more than once in tissue culture. The peripheral leukocyte system will be used in looking for biologic effects of high-energy radiation in tests with outer-space rockets.

An identification study of host and donor antibody-forming cells in heterologous chimeras sensitized to sheep red blood cells showed both host and donor activity.

A structure in lymphatic tissue known as the tingible body seems to be an important site of granulocyte accumulation and destruction in normal animals given large doses of rat bone marrow. This change is one of the earliest detectable events in the development of immune tissue reactions.

TRAINING, EDUCATION, AND INFORMATION PROGRAM

International Reactor Program at ORNL. — Forty-eight students currently enrolled in ORSORT programs have completed 29 weeks of school since the beginning of classes in November 1960.

Plans have been made for conducting a fourth session of foreign programs, starting in September 1961. Brochures announcing this program have been distributed to industrial and governmental agencies in 45 countries.

ASEE-AEC Basic Summer Institute. — Plans have been made for presenting a series of ORSORT experiments in physics and radiochemistry to participants of the 1961 ASEE Basic Summer Institute conducted by North Carolina State University during the week of August 7–11, 1961.

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