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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

JANUARY 1961

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

JANUARY 1961

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OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee
operated by
UNION CARBIDE CORPORATION
for the
U.S. ATOMIC ENERGY COMMISSION

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

January 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.

SPECIAL NUCLEAR MATERIALS PROGRAM

Radiochemical Processing Pilot Plant. — Approximately 1 kg of plutonium flushed from the equipment in Building 3019 was recovered by anion exchange in Building 3505. The plutonium was separated from fission product gamma emitters by a factor of 8×10^3 and was concentrated by a factor of 50 with one anion exchange cycle.

Cleanup of contamination in Building 3019 was continued; painting of cell 3 was completed. Dry-stacked shielding blocks were removed from cells 5, 6, and 7, and all process equipment and cell walls were washed with various decontamination reagents, using a high-pressure solution spray system.

Cells 5 and 6 were washed with approximately 100,000 gal of aqueous solution, decreasing the transferable alpha contamination on most cell and equipment surfaces to less than 10^4 dis/min per 100 cm^2. Decontamination of surfaces within the cell will continue in an attempt to decrease the transferable alpha contamination level to 3000 dis/min per 100 cm^2 before painting.

REACTOR DEVELOPMENT PROGRAM

THERMAL-BREEDER REACTOR PROGRAM

Homogeneous Reactor Program

Homogeneous Reactor Test. — On January 4 the HRT attained a full power of 5 Mw (1400 psig, 270°C core, 230°C blanket) for the first time since the core flow was modified (fuel solution now flows through the core vessel from top to bottom). No indications of fuel instability could be detected during the 90 hr at the high-power level. Power oscillations, which have been "rougher" since the flow was reversed, ranged up to about 20% of the power level while at 5 Mw. However, oscillations larger than 15% of the power level occurred, on the average, only four times per hour.

One of the fuel feed-pump heads failed. Operation was continued, using the spare head.

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The reactor was refueled, since studies of five-component models of the chemical system indicated that the nickel concentration of the old fuel (initially charged in March 1958) was reaching a level which could cause nickel-salt precipitation. High reactor temperature and pressure (subcritical) were maintained while the old fuel was diluted out of the high-pressure system, concentrated to a heel in the dump tanks, and transferred to the outer storage tanks. New fuel was charged to the system during this procedure. The entire operation took only three days. The run designation was changed from "23" to "24" to denote the change in fuel.

The initial step after the new fuel was charged was to pretreat the high-pressure system with oxygenated heavy water and then with subcritical fuel at 280°C to ensure that a protective film (against corrosion) is being maintained.

Internal radiolytic-gas recombination tests were the final experiments conducted with the old fuel and the initial experiments with the new. The apparent copper activity was 1.5 to 2 times higher than that predicted on the basis of out-of-pile experiments; this high activity has been observed consistently in the HRT.

Operation is being resumed at full power with the new fuel charge. Studies of the reactor behavior will continue.

HRT Chemical Pilot Plant. — Solids collection rates with the revised multiple hydroclone have leveled out at 0.3 to 0.4 g/hr; they are not significantly different from those obtained with a single hydroclone. Improvements which result from recycling the multiclone underflow-receiver contents through the collection hydroclone appear to be completely offset by a lowering of the multiclone efficiency when it is operated as at present with induced underflow.

Two additional tests of the UO_4 fuel-processing system were made with 4-kg batches of natural uranium. Uranium loss on the first of these tests was only 3.6%, compared with 6.3% on a previous test. Efforts will be made to reduce the losses even further by recycling the initial filtrate, which contains higher than normal concentrations of uranium. A decontamination factor of 8 from copper and nickel was achieved without washing the precipitate; three washes increased the decontamination factor to over 100. The process continues to appear promising as a method for limiting the accumulation of nickel in the fuel of large-scale reactor systems.

Thorium Blanket Studies. — In studies of the preparation of thoria-gel particles larger than 1/8 in. in diameter, spheres were made from sols of thorium oxide prepared by steam denitration of thorium nitrate by further loading of 1 ml of sol with 2 to 2.75 g of steam-stripped oxides which had been calcined in air at 25 to 350°C, followed by manual forming to spheres. On drying, these spheres tended to crack into quarters but did not further crack on calcination to 1200°C. Electron micrographs at 20,000× magnification of carbon replicas of uncracked and freshly cracked surfaces of gels prepared by evaporation of unloaded sols showed that larger gel particles are composed of grains of about 0.67 μ diameter. Cracks, when present, occurred at the grain boundaries, but few traversed a grain. Binding forces between grains appeared to be significantly weaker than those within the grain.

Reactor Analysis. — Statistical studies of digitalized power data from the HRT are being made with the aid of two IBM 7090 programs. One program computes the frequency distribution of power peaks, as well as the standard deviation from average power. The other program is used to compute the reactivity addition which corresponds to the observed power trace. Although not enough data are available to draw satisfactory conclusions, it appears so far that reduction in pressure increases the average size of power deviations. The effects of changes in operating temperature and fluid flow rate are as yet uncertain. The number of fluctuations per unit time increases with power level, as does the magnitude.

Calculations of the temperature and concentration effects which follow a rise in HRT power (from heat loss to 5 Mw) are in progress. Calculations show that a small initial temperature rise is to be expected (about 1°C), followed by a gradual fall to about 1°C below the temperature at heat-loss power.

Metallurgy and Ceramics. — In support of the effort to make a thorough temperature scan of the HRT core vessel, it was necessary to effect a closure between the 1/8-in. thermocouples and a 1-3/4-in.-thick flange. Twelve thermocouples sheathed with type 310 stainless steel penetrate the type 347 stainless steel flange on about 1/2-in. triangular centers. Only a 2-in.-diam area is available for all these penetrations. The closure must contain the fuel solution and/or vapor at temperature (up to 260°C) and pressure (up to 2000 psi). A modification of the MSRP tube-to-tube-sheet closure procedure was used. The sheaths were Heliarc-welded to the flange on the outside by using a trepan joint. The assembly was then back-brazed with 82% Au–18% Ni alloy, which has shown superior corrosion resistance in HRT fuel environments. The powdered braze metal was placed in a trepanned sump around each couple and added to the braze joint at a point about 1/2 in. from the inside surface by three feeder holes.

A full, smooth braze fillet was obtained, and the joint, after helium leak-checking, was considered acceptable for the intended application. The disparity between the couple size and the flange thickness was greater than for any other assembly to which this method of closure has been applied.

Out-of-Pile Solution Corrosion Tests. — The corrosion resistance of Zircaloy-2 and titanium 75A to high-temperature uranyl sulfate solutions was investigated in a small high-pressure titanium pump loop. In the first experiment, specimens of the two alloys were exposed to a heavy-water solution containing 0.04 m UO_2SO_4, 0.02 m $CuSO_4$, and 0.035 m D_2SO_4 at temperatures between 325 and 360°C for 165 hr. During most of this run the circulating fluid was composed of two different liquid phases. At flow rates between 7 and 34 fps the Zircaloy-2 specimens developed thin black films, small areas of which were beginning to transform to a white oxide. All specimens showed weight gains of about 0.1 mg/cm^2. Titanium specimens developed thin black films and showed insignificant weight changes.

The same specimens were then exposed at 360°C for 563 hr to a solution containing 0.085 M UO_2SO_4, 0.02 M $CuSO_4$, and 0.245 M D_2SO_4. This solution is chemically stable to at least 365°C and at that temperature contains about 10 g of uranium per liter. At the end of this run the Zircaloy-2 specimens were covered with a whitish-gray oxide and showed weight losses

which corresponded to corrosion rates between 6 and 7 mils/yr. The titanium specimens did not change in appearance or weight as a result of the high-temperature exposure.

These results show that fuel solutions stabilized with relatively high acid concentrations can be satisfactorily contained in titanium vessels at temperatures as high as 360°C, but they indicate appreciable corrosion of Zircaloy-2 in such systems.

Out-of-Pile Blanket Materials Tests. — Two samples of experimental thorium-uranium oxide (10 wt % UO_2) microbeads which were prepared at Argonne National Laboratory were circulated in 300-hr toroid tests to evaluate their integrity and erosiveness. The tests were made at 280°C and 26 fps at a concentration of 500 g of Th-U per kg of D_2O, with 100 psig oxygen overpressure. The preparations were degraded from average particle sizes of 15 to 8.8 μ and 49 to 32 μ respectively. Excessive attack was noted on specimens of type 347 stainless steel, titanium, and Zircaloy-2 which were exposed to the slurry of smaller sized material. Tenacious films of slurry solids coated the specimens exposed to the slurry of the larger sized microbeads.

In-Pile Blanket Materials Tests. — For in-pile slurry loop samples, apparent mean crystallite sizes originally greater than 2000 A, as determined by x-ray line broadening, decreased after the beginning of irradiation to 100–300 A. Since lattice defects as well as particle degradation could give such a result, annealing experiments have been initiated. No effect of annealing was noted at 400°C, the initial temperature of the series.

Evaluation Studies

Thorium-Breeder-Reactor Evaluation Studies. — Investigation was made of the nuclear benefits resulting from the addition of beryllium to a gas-cooled, graphite-moderated reactor. The graphite case (containing no fuel) of the movable fuel element was progressively replaced with BeO. The breeding gain increased with BeO content until it was 0.02 greater than its initial value; further increase in BeO content resulted in a decrease in breeding gain. The increase in breeding gain would be largely offset eventually by losses to Li^6 resulting from the (n,α) reaction in beryllium, unless the fuel cases were processed to remove the Li^6; such processing would probably not be economical. The replacement of graphite by beryllium metal instead of BeO gave nearly the same results as those obtained for BeO addition.

The loss of neutrons associated with shifting of protactinium at a finite rate rather than at an infinite rate was estimated for an aqueous-homogeneous breeder reactor. This loss relative to that associated with an infinite rate of blanket shifting was termed the "excess loss." In a blanket in which the fertile material was shifted radially among three different zones, the excess loss was only 0.25% if the shifting were done daily, 1.2% with a three-day holdup, and 5.2% with a ten-day holdup prior to shifting.

MOLTEN-SALT REACTOR PROJECT

MSRE Design. — The volume fraction to be occupied by fuel in the MSRE core was increased to 22.5% with some reduction of the critical mass and fissionable inventory of the reactor. Fabrication drawings of all graphite pieces for the core are being checked.

The hydrodynamics of several locations in the core were studied. The graphite support grating appears to produce sufficient pressure drop to override the radial pressure gradient in the bottom plenum. The flow passage between the INOR-8 shield and the dome will be enlarged in order to minimize the pressure drop in this region. The hole distribution in the constant-area inlet header of the core was determined experimentally and incorporated into the design.

Drain-tank drawings are being revised after the first round of comments. Present plans call for two identical cooled tanks for the fuel salt, a secondary-salt tank, a flush-salt tank, and a spent-fuel storage tank.

Detailing of the radiator enclosure and superstructure is nearly complete. The design calls for an insulated and heated casing with counterweighted sliding doors. The doors are held open by magnets and can be closed by either free fall or powered descent.

The primary-cell heater circuits have been designated, and electrical design is proceeding.

Structural design drawings of the storage pits and of the shielding slabs for the primary vessel are being prepared. Design layouts of the filter pit, filter-pit ducting, drains, sump pump, and primary-vessel air evacuation system are being prepared.

Drafting was completed and drawings are being checked for the pump-flange torquing tool, the 6-in. freeze-flange torquing tool, and the 30-ton to 7-ton crane hook reducer. Specifications and drawings for the windows in the maintenance control room were distributed. Specifications for the General Mills manipulator were sent out for comments.

Design of the sampler-enricher is essentially complete.

The MSRE preliminary hazards report is being reviewed and is scheduled for issue in February.

Component Development. — Five corrosion pump loops of INOR-8 have operated with various fluoride salts at 1300 to 1500°F for periods of from 7000 to 20,000 hr. Two loops, which began operation in October 1958, were dismantled for metallurgical examination after 20,000 hr each.

Piping of the first configuration of the engineering test loop was completed, and instruments are being installed.

The test of the frozen-lead shaft seal pump was terminated after 22,000 hr when the pump ceased operating. The 3/16-in. shaft jammed and was broken when attempts were made to free it. There was visual evidence of severe corrosion of the shaft and container in the vicinity of the lead.

Velocity measurements with the latest volute design for the one-fifth-scale core model indicate that the flow-rate distribution is uniform at the rated flow (50 gpm) and at 40% flow (20 gpm). The fluid velocity at the bottom of the vessel and between the graphite container and the pressure vessel was found to be uniform around the vessel.

An Inconel metal-alloy freeze valve was tested, and it failed because of a lack of wetting of the Inconel surfaces at 1500°F by the 72% Ag–28% Cu solder alloy (mp, 1434°F). (INOR-8 is wet by this solder.)

The direct resistance-heated and replaceable sheathed resistance-heated freeze valves have operated successfully for 86 and 34 cycles respectively. Test units for the pipe and core-vessel heaters are being constructed.

Remote Maintenance. – The part of the remote-maintenance demonstration area that is serviced by the manipulator is being cleared to provide space for construction of MSRE problem mockups. The first problem, a mockup of the primary pump, was constructed to duplicate the bolt extensions and auxiliary piping. Installation in the remote-maintenance demonstration area was started.

The primary-salt system and the containment vessel of the one-twelfth-scale model of the MSRE are almost complete, and construction of the model building has started.

A preliminary listing of maintenance tools and equipment was completed. Performance data from the single-seal two-block cone-seat disconnect and from the buffered integral ring-joint disconnect using spring clamps indicate that at least five satisfactory makes and breaks are possible. Each seal is subjected to a minimum of three consecutive 300°F thermal cycles after each make-and-break test. Work was started on manifold disconnects with a single buffer region.

Pump Development. – The pump with a molten-salt-lubricated journal bearing has now operated for 6600 hr with $LiF-BeF_2-UF_4$ (62-37-1 mole %) at 1225°F. The Inconel centrifugal pump (PKP type) has now circulated $LiF-BeF_2-ThF_4-UF_4$ (65-30-4-1 mole %) at 1225°F for 2880 hr. Equipment modifications were nearly completed for the back-diffusion experiment with Kr^{85}.

The time required to reduce the concentration of dissolved CO_2 in water by a factor of 2 has been tentatively determined, for the best stripper configuration tested to date, as 1.3 times that required for an ideal stripper. Testing of a configuration which increases stripper flow to nearly 100 gpm is planned; this is believed to represent the maximum stripper flow rate that can be controlled in the present primary-pump tank.

Evaluation of thermal-stress calculations indicated the need for including the effects of the radial force and the bending moment from the spherical shell and the restraint at the top end of the cylindrical portion of the pump-tank vessel. A refined calculation including these effects is being made based on an estimated reasonable temperature distribution in the vessel.

Fabrication of the hot-test MSRE primary pump progressed satisfactorily. Initial reviews were made of the flowsheets for the gas and lubricant systems. Mechanical design of the primary pump was nearly completed; work is still to be done on the details of the stripper configuration, the heat sink for the internal nuclear shield, and the air cooling channel for the pump-tank wall.

Chemistry. – The behavior of the MSRE fuel, $LiF-BeF_2-ZrF_4-ThF_4-UF_4$ (70-23-5-1-1 mole %), as it begins to freeze was explored further by examining quenched samples of systematically altered compositions. Variations

in the $LiF:BeF_2$ proportions in a mixture containing fixed proportions of ZrF_4-ThF_4-UF_4 (7-1-1 mole %) gave melting points changing from 470°C at 16 mole % BeF_2 to 446°C at 25 mole % BeF_2; between 16 and 22 mole % BeF_2 the primary phase is a ternary compound of LiF, BeF_2, and ZrF_4, but Li_2ZrF_6 is the primary phase at 25 mole % BeF_2. At temperatures which depend on the composition but which are near 435°C, where but little crystallization has occurred, Li_2BeF_4 appears as the second phase to crystallize, followed by a solid solution, $7LiF \cdot 6(U,Th)F_4$, at about 430°C, where but little liquid remains.

A melt of LiF-BeF_2 (63-37 mole %), closely resembling the MSRE coolant, LiF-BeF_2 (66-34 mole %), contains 1000 ppm oxygen when saturated with BeO. This corresponds to a solubility product $[Be^{++}][O^{--}] = 5 \times 10^{-4}$. Preliminary results also indicate that 600 ppm oxide ion corresponds to saturation with UO_2 in an MSRE-type fuel (0.5 mole % UF_4) which contains no ZrF_4. Hence, ZrO_2 precipitation from the MSRE fuel could be expected at an oxide ion concentration somewhat lower than 600 ppm.

The solubility of HF in LiF-BeF_2-ZrF_4-ThF_4-UF_4 (65-28-5-1-1 mole %) follows Henry's law and ranges from 17×10^{-6} to 8×10^{-6} mole of HF per cubic centimeter of fuel per atmosphere of HF pressure between 500 and 700°C, in accordance with a heat of solution of −5.6 kcal/mole.

The absorption spectrum of UF_4 in the MSRE fuel was observed at 528°C over the wavelength range between 300 and 2500 mμ. Relatively strong absorbance maxima were found at 1000 mμ, with a shoulder at 890 mμ, and at 640 mμ. A broad absorbance was found in the region from 370 to 550 mμ, with two distinguishable maxima at 516 and 470 mμ. This pattern is similar to but not identical with the spectrum of UF_4 in other molten-salt solvents used earlier.

Metallurgy. – Additional data were obtained on the mechanical properties of INOR-8 for use in the design of the MSRE. Design values were established for the alloy through 1400°F.

The effect of annealing temperature on the creep properties of INOR-8 was studied in creep tests at three stress levels where the specimens had been given a prior annealing treatment at 2150 or 2250°F. The tests indicated that the annealing temperature has no significant influence on the minimum creep rate at 1300°F and at the stress levels tested.

Stress-rupture tests on notched specimens of INOR-8 were made at 1300°F at stress levels from 12,500 to 25,000 psi. The results obtained to date show no tendency toward notch sensitivity.

In-Pile Tests. – The third MSRE graphite-fuel capsule is being assembled. The fuel, which is now being prepared, will differ from the MSRE fuel in that it will contain 1.5 mole % UF_4, as compared with 1 mole % UF_4 for the MSRE fuel. The additional UF_4 is needed so that a power density of 200 w/cc can be obtained in the experiment.

Present plans call for an out-of-pile test and subsequent examination of the parts prior to the final assembly of the experiment. Unless the preliminary test reveals some unforeseen problem, the experiment will be inserted in the MTR on April 10, 1961.

Reactor Analysis. – Nuclear calculations were completed during the month for the currently proposed MSRE core, with a fuel volume fraction of 22.5%. The proposed control tubes (ORNL-3062, p 6) have an estimated

combined reactivity worth of 13% $\delta k/k$ in the clean, hot condition. The reactivity increase resulting from filling 7% of the core graphite with fuel salt was estimated to be 6%; so the four tubes provide an adequate amount of control in case fairly rapid graphite penetration should occur (a remote possibility).

Three-group, two-dimensional cell calculations were done to determine the shape of the neutron flux above 0.1 Mev within a core graphite block; the results indicate that there is about a 2% flux depression from the inner edge of a fuel channel to the center of the block, and thus differential shrinkage from this source should not be serious. However, differential shrinkage due to the nonuniform flux shape across the reactor will be very nearly the same as previously estimated for a core with 12% fuel volume fraction (ORNL-3014, p 32).

GAS-COOLED REACTOR PROJECT

Instantaneous Fission-Gas-Release Experiment. — The sample of UO_2 now being irradiated is in the form of thin plates sandwiched together. The two UO_2 slabs are sandwiched between two ThO_2 plates to ensure that there will be no fission-recoil products embedded in the metal walls of the capsule which could subsequently diffuse out and contribute to the temperature-dependent gas release. The fission-gas release from this ThO_2-shielded sample is essentially the same as for the previous non-shielded samples, demonstrating that back-diffusion of fission products from the metal capsule is not a significant contribution to the temperature-dependent fission-gas release.

A series of measurements is in progress to determine the rate of release of iodine from the UO_2 as a function of temperature. These measurements are necessarily time-consuming, since equilibrium must be obtained between the iodine decaying and being deposited. This may take several days, depending upon the initial amount of iodine present in the system. One measurement at 720°C revealed that roughly 100% of the Xe^{135} emerged from the UO_2 in the form of I^{135}. This is not surprising, since recoil release predominates at this temperature. A second measurement at 890°C showed that 80% of the Xe^{135} emerged from the UO_2 in the form of I^{135}. The total release of isotope-135 increased by a factor of 3.5 between 720 and 890°C. These measurements are continuing so that the temperature dependence of iodine and xenon release can be established.

Beryllium Irradiation Experiments. — Density measurements have been completed on beryllium specimens irradiated in the third high-temperature in-pile swelling experiment. There was negligible density change of the specimens irradiated at 600°C or lower. At higher temperatures some swelling occurred.

Two in-pile tube-burst experiments are now complete except for post-irradiation examination of the specimens. Six specimens irradiated in the ORR poolside position and eight specimens irradiated in the core position were pressurized after 34 days of reactor operation at 30 Mw. This is an exposure of approximately 8×10^{19} neutrons/cm^2 for poolside specimens and 2.4×10^{20} neutrons/cm^2 (above 1 Mev) for specimens in the core

position. There is a great deal of scatter of both the out-of-pile and in-pile data, but a serious reduction in time to rupture is indicated.

Evaluation of Mixed Oxide Fuels. — The fission-gas retention properties of a BeO-based mixed oxide fuel were determined in the temperature range 910 to 1400°C. Data indicate that below 1200°C there was a rapid initial release of xenon that amounted to about 0.01% of the xenon generated based on burnup calculations and did not vary with temperature. After the initial release, no diffusion release was detected at temperatures below 1200°C. However, at 1400°C the data indicate that the initial rapid release was followed by the usual diffusion-controlled release exhibited by UO_2 and $BeO-UO_2$.

It has been reported that thermal cycling of mixed oxides and UO_2 causes additional fission-gas release similar to that occurring during initial heatup. Eight thermal cycles were run between 200 and 910°C to see whether any xenon would be released as a result of cycling. No additional release was detected.

Welding of Ferritic Steels to Stainless Steels. — A welding procedure was developed and qualified in accordance with Section IX, ASME Boiler and Pressure Vessel Code, to cover the tungsten-arc welding of ASME P-Number 1 carbon steel materials to stainless steels with BP-87 alloy filler wire. The mechanical properties of metallic-arc welds of this general type made with BP-85 filler metal were studied at room and elevated temperatures. Transverse tensile specimens were machined from typical weldments, and tests were run at temperatures representative of the maximum service temperature for each type of joint. All specimens of ASTM A-212 grade B steel welded to type 304 stainless steel were stress-relieved for 4 hr at 1150°F, and several of these specimens were aged for 1000 hr at 650°F before testing. Specimens of ASTM A-387 grade D steel welded to stainless steel were stress-relieved for 1 hr at 1350°F. In every case the specimens failed in the base metal after an elongation of about 20%. The tensile strengths were essentially the same as those for the base materials at the test temperatures.

Adsorbers for Fission Product Gases. — Iodine-retention tests were performed using 0.25-in.-deep adsorber beds of various materials to give a rapid indication of the relative value of the adsorber material for application to full-scale iodine adsorbers with much larger depths of adsorbent material. A bed of Pittsburgh PCB coconut charcoal, 6—16 mesh, was found to have retained 97.6% of the iodine initially present after 168 hr at 325°C. Previous tests in this series on Columbia G charcoal, Pittsburgh BPL charcoal, and silver-plated copper mesh gave efficiencies of 94.7, 16, and 95.7% respectively. A sample of Whetlerite charcoal, type ASC, 12—30 mesh, impregnated with salts of copper, chromium, and silver, is being tested. This charcoal is designed for use in gas masks for protection against war gases and is available commercially. The test has been in operation for 150 hr, and no evidence of iodine release has been obtained.

GCR-ORR Loop No. 1 Operation. — The two regenerative compressors installed in the twin-turbine vessel have operated satisfactorily for a total running time of 2100 hr. The compressor operation has been over a range of temperatures up to 600°F and speeds from 6000 to 12,000 rpm, but

the loop pressure has been limited to 45 psia, pending approval for operation at higher pressures. Several material irradiation runs were made with the compressors operating in series at 12,000 rpm at reduced temperature to attain the desired mass flow.

HIGH FLUX ISOTOPE REACTOR

Physics. – Control-plate materials for the second HFIR critical experiment (HFCE-2) have been selected, and it is anticipated that they will also be used in the actual HFIR core. The particular materials selected are aluminum (white region), nickel (gray region), and silver clad with nickel (black region). The nickel and silver replace the previously selected tantalum or tungsten dispersions in aluminum for the gray region and europium or dysprosium dispersions in aluminum for the black region. A lack of success in producing satisfactory dispersions and the relatively high cost were responsible for the substitution of nickel and silver. In addition, these latter materials burn out at a significantly lower rate, allowing a control-plate life in excess of one year. The disadvantages are higher heat generation rates, lower thermal conductivity (nickel vs aluminum), greater weight, and the necessity for a mechanical joint between the nickel and the aluminum regions.

In preparation for critical experiments using actual HFIR fuel elements, calculations were made for determining the neutron multiplication of the fuel elements when completely submerged in water. Results indicate that the complete core, containing burnable poison (in the inner annulus) and 8.01 kg of U^{235}, will be subcritical by about 0.02. The outer fuel annulus alone, containing no burnable poison and over two-thirds of the fuel, was subcritical by 0.25.

Control and Instrumentation. – The title I design of the nuclear and process control and instrumentation has been established, and the drawings and criteria have been completed.

The development of the over-all system is in progress in several areas; namely, experiments conducted in the Pool Critical Assembly which were aimed at establishing the neutron flux level at the proposed safety ion chamber locations indicate that a neutron flux of approximately 5×10^7 thermal neutrons cm^{-2} sec^{-1} will exist at the chamber location. It will therefore be necessary to augment this flux by incorporating a collimator between the beryllium reflector and the pressure vessel in order to raise the flux to at least 5×10^{10} neutrons cm^{-2} sec^{-1} in the space provided.

Test prototypes of the newly developed multistage ionization chambers which are to be used in the safety system are being assembled.

Tests are being conducted on a fast-release three-coil electromagnet along with its associated driver amplifiers which are to be used in the "two-out-of-three" coincidence system. Release times of about 5 msec have been achieved with the magnet supporting 250 lb with a 500-lb force.

Corrosion. – Two tests which were designed to investigate the effect of system pressure on the corrosion of 6061 aluminum subjected to a high heat flux have been completed. The conditions were the same in both tests,

except in the first the pressure on the system was 70 psi, and in the second it was 900 psi. The other conditions were: heat flux, 2×10^6 Btu hr^{-1} ft^{-2}; water pH, 5 (HNO_3); inlet water temperature, 173°F; outlet water temperature, 219°F; flow rate, 40 fps; test duration, 10 days.

During the first test (70 psi) the maximum observed rate of temperature increase of the specimen (due to the buildup of an oxide film on the water-cooled surface) was about 3°F/day. The thickness of metal removed as a result of corrosion was constant throughout the length of the specimen at 0.2 to 0.3 mil, except at one location very near the specimen outlet where the penetration was 0.5 mil. During the second test (900 psi) the maximum rate of temperature increase was 17°F/day. The thickness of metal removed as a result of corrosion increased nearly linearly from 0.3 mil at the specimen inlet to 0.9 mil an inch from the outlet (5.5 in. from inlet). In the last inch, corrosion damage was more severe, and in one location a penetration of 3.9 mils was observed.

The results of these two tests indicate that the temperature of the aluminum was lower and the extent of penetration was less at the lower pressure than at the higher pressure. At the lower pressure, boiling on the aluminum surface was possible. Whether or not boiling did occur, and if it did, whether boiling alone was responsible for the observed difference, must be determined in subsequent tests.

Metallurgy: Fuel-Element Core Preparation. — Six ingots of 24.2 wt % U–Al alloy were cast for use in fabricating the HFIR critical-experiment outer fuel annulus. These were extruded into rod and analyzed chemically at three locations for uranium content. The average analysis of these castings was found to be 24.7% U, with a minimum and maximum of 23.5 and 26.5 wt %.

Approximately 100 fuel cores were forged to a shape required for plate rolling. These were made from sections cut from the extruded rod but adjusted in volume to result in a U^{235} content of 15.4 g. Densities of all cores are being recorded.

Metallurgy: Fuel-Plate Fabrication. — Approximately 40 plates for the critical assembly were rolled to finished dimensions. The plates were blister-annealed at 500°C, and only "fit blisters" that had developed during hot rolling were observed. These were found in the regions of the plates at the ends of the core.

Ultrasonic equipment has been converted to allow testing of the fuel plates for lack of bonding. Longitudinal scans of 150 to 200 in./min can be accomplished with this system, and the nonbonded area will be determined by the amount of indexing or transducer motion between successive longitudinal scans. Calibration studies indicate that 1/16-in.-diam nonbonded areas can be readily detected. Preparations are being made to ultrasonically test 150 of the 600 plates being made for the critical assembly.

Metallurgy: Fuel-Element Assembly. — A temporary welding shop has been set up for the fabrication of the critical assembly. A complete mark II-A mockup inner fuel annulus, consisting of half-length dummy fuel plates, has been assembled, and preliminary welding tests are in progress. The welding tests have been designed to (1) give information concerning the utility of end combs and preplaced Teflon spacers for preweld plate alignment and (2) qualify the welder assigned to fabricate the critical element.

An investigation of automatic consumable-electrode welding methods has been continued. However, it is presently felt that the investigation will not go beyond a preliminary testing stage until after the critical assembly has been completed.

Design: Reactor. – The conceptual design report on the reactor core and pressure vessel was completed and was issued for inclusion in the HFIR title I report being prepared by the architect-engineer.

Specifications and drawings, giving the design criteria, were issued for purchase of the reactor pressure vessel, the primary heat exchangers, and the primary circulating pumps.

Detail design of the reactor core components is in progress.

Drawings of the fuel element and the beryllium are partially complete. Their supporting structure is ready for detailing.

The reactor control drive design, which is similar to the ORR drive concept, is being detailed. Drive parts will be built by use of these drawings and will be tested in the HFIR mockup to determine the drive reliability.

Analytical studies have been completed to determine the reactor and clean pool criteria. The effect of spent fuel storage on heat removal and shielding was considered in the studies.

Design: Critical Experiment. – The design of the critical experiment (No. 2) equipment is completed. The experiment is designed to simulate the reactor in such a manner that the following measurements can be made: power distribution, control region variation worth, fuel worth, temperature coefficients, and void coefficient.

MARITIME REACTORS PROGRAM

Physics. – A uniformly enriched egg-crate core containing 4.2 wt % stainless steel was compared with a uniformly enriched egg-crate core containing 3.0 wt % zirconium at 68 and 508°F in the NS "Savannah" core I design. The calculations indicate that the temperature deficit for a Zircaloy egg-crate core is only 0.004 Δk greater than for the stainless steel egg-crate core. A dependence of the temperature deficit on the egg-crate material was expected because this material is in a high thermal-neutron flux region and because the thermal-neutron flux distribution changes with core temperature.

Irradiation Tests. – Experiment No. 3 fuel specimens are now receiving further irradiation in the ORR loop. At present they have completed two ORR cycles at 16 Mw and one at 30 Mw. Experiment No. 1 fuel specimens have been received at the Vallecitos facility of the General Electric Company for postirradiation examination.

ARMY PACKAGE POWER REACTORS

Metallurgy. – Studies for defining the mechanism of boron loss during sintering of UO_2–stainless steel fuel compacts containing boron-bearing

burnable poisons have been essentially completed, and the loss has been measured quantitatively. The rate of loss is directly proportional to the rate of change of total compact surface area. The mechanism of the loss appears to be the synthesis of a boron-hydrogen species, but formation of the compound is not the rate-controlling process. Rather, the total loss is controlled by parameters which affect the rate of sintering of the compact, such as water content of the hydrogen, sintering temperature, and initial compact densification.

The rate of boron loss from 0.128 wt % boron—stainless steel sheet heat-treated at 1135°C is controlled by the rate of boron diffusion to the gas-metal interface. The loss is independent of the reacting species available (i.e., the sintering environment).

Postirradiation examination of all iron-boron absorber specimens from the ORNL-MTR-28 irradiation test program has been completed except for metallographic examination and burnup analyses. Present results indicate that only specimens subject to estimated B^{10} burnups of less than 4% remain dimensionally stable. Specimens subjected to higher burnups showed complete loss of core-to-cladding bonding, with subsequent blistering. Thickness increases of 20 to 30% were noted for specimens subjected to estimated B^{10} burnups in the 20 to 30% range. Two groups of three specimens (low, medium, and high burnup) were subjected to postirradiation annealing at 570°F. One group was annealed at atmospheric pressure, the other in an autoclave at 1200 psi. Neither of the low-burnup specimens exhibited any dimensional changes as a result of this heat treatment. The four remaining specimens showed large thickness increases as a result of increased blistering, and little effect was produced by the application of external pressure during the annealing operation.

Irradiation of five miniature Eu_2O_3—stainless steel dispersion absorber elements from the ORNL-MTR-39 program has been completed. Postirradiation examination has been conducted on these specimens, with the exception of metallographic examination and burnup analyses. All five specimens remained dimensionally stable and structurally intact up to exposures of 12.1×10^{20} thermal neutrons/cm^2 (unperturbed).

ANP PROGRAM

Shielding Research. — The new Tower Shielding Reactor (TSR-II) was operated at power levels of up to approximately 30 kw with full water flow of 800 gpm. The pressure drop across the reactor was approximately 31 psig. No major difficulties were encountered, and it appears that the power level can safely be increased to 100 kw and finally to 5 Mw. The reactor was operated in the kilowatt range 16 hr/day for three weeks with planned shutdowns at about 15-min intervals. The neutron leakage from the pressure vessel was mapped.

Experiments conducted in the laboratory showed that Borester 7, an organic ester containing boron, can be mixed with transformer oil to give a carbon-to-hydrogen ratio that closely approximates that of polyethylene containing 2.6% boron. This mixture will be used in the Boeing crew shield to simulate plastic during testing of the Pratt and Whitney shield mockup with the TSR-II. The preanalysis was completed for the experiments to be

conducted at the Tower Shielding Facility with the Pratt and Whitney shield mockup and the Boeing crew compartment.

Columbium Alloy Research. – The reaction rates of Cb–2% Sn and Cb–1% Pd with oxygen at a pressure of 5×10^{-4} mm Hg and a temperature of 1832°F were shown to be similar to the reaction rates of unalloyed columbium under the same conditions. The reaction rate of Cb–25% Ti–5% Al with low-pressure, high-temperature oxygen was shown to be three times as great as that of unalloyed columbium; this alloy reportedly oxidizes only a hundredth as fast as columbium in air at 1 atm pressure and 1832°F.

Two types of composite oxidation-resistant tubing consisting of stainless steel over columbium were received from Superior Tube Company for metallographic examination, nondestructive testing of the bonding, and joining studies. One type of tubing consists of type 446 stainless steel over columbium and the other of type 304 stainless steel over copper over columbium. Composite tubing is also being prepared from Cb–1% Zr alloy, and sheets of this alloy are being clad with aluminum bronze.

Tee joints were prepared from stainless steel and columbium with 13 types of brazing alloy in an evaluation program. The stainless steel was not wet by alloys consisting of 68% Ti–28% V–4% Be or 80% Zr–17% Fe–3% Be, and, as a result, the joints made with these alloys fractured when thermal stresses set up upon cooling. Other brazing alloys are being evaluated further. The flow points determined for the various alloys are given below:

Brazing Alloy Composition (wt %)	Flow Point (°C)
67 Zr–29 V–4 Fe	1300
60 Zr–25 V–15 Cb	1300
95 Zr–5 Be	1000
65 Zr–25 V–10 Cb	1280
48 Ti–48 Zr–4 Be	1050
63 Ti–27 Fe–10 Mo	1250
63 Ti–27 Fe–10 V	1300
68 Ti–28 V–4 Be	1250
47 Ti–47 Zr–3 V–3 Be	1150
45 Ti–40 Zr–15 Fe	1050
76 Zr–20 Cb–4 Be	1050
80 Zr–17 Fe–3 Be	1000
62 Ti–26 Fe–8 Mo–4 Zr	1250

A new tentative theory was developed and is being explored which reconciles the data obtained on the aging of wrought Cb–1% Zr alloy with the

data obtained on welded material. The concept of oxygen solubility in the Cb–1% Zr alloy appears to explain most of the data. Briefly, the theory assumes that the solubility of oxygen in the alloy decreases with temperature to only a few hundred parts per million at about 1400°C. Precipitation of zirconium oxide could then explain the aging reaction. Some assumptions about particle sizes must be made to explain the lack of aging in high-oxygen-content alloys, but these assumptions can be made reasonably. Experiments are being designed for testing the theory.

The first of a series of in-pile experimental assemblies containing tube-burst-type specimens of a Cb–1% Zr alloy was removed from the ORR poolside facility. No time-to-rupture measurements were obtained during the irradiation because of faulty brazed joints and undersized heaters. Inspection of the irradiated and stressed specimens will, however, provide some strain data, information on contamination of the tubes by impurities in the helium atmosphere, and data on activation of the tubes. The degree to which the tubes have become radioactive will influence the chemical analysis procedures. A second assembly of Cb–1% Zr alloy tube-burst specimens with improved brazing and heavier-duty heaters was installed in the ORR. In this assembly, pressures up to 2500 psi will be used with the tubes at 1800°F. Out-of-pile testing at this pressure gives a time to rupture of approximately 100 hr.

Liquid-Metal Technology. – In a comprehensive effort to determine the relative compatibilities of iron-, nickel-, and cobalt-based alloys with boiling potassium, the construction of a Haynes alloy No. 25 system for circulating boiling and condensing potassium was nearly completed. This system will operate under the same conditions as the type 316 stainless steel test loop No. 2, namely, 1600°F boiler temperature and 50 fps vapor flow rate. A Cb–1% Zr alloy loop is also being fabricated in which boiling potassium will be circulated.

Installation of the liquid-metal boiling-heat-transfer apparatus was essentially completed. The initial test section is a 40-in. length of 1-in.-OD, 0.065-in.-wall tubing, with associated heaters (4 kw/ft) and surface thermocouples. The maximum flux attainable with this arrangement is calculated to be about 65,000 Btu hr^{-1} ft^{-2}. Although this is considerably below conservative burnout-flux estimates, high exit qualities of the potassium vapor can be achieved at the lower flux rates. A second boiler section capable of surface heat fluxes of 500,000 Btu hr^{-1} ft^{-2} is being fabricated.

Beryllium Oxide Research. – Phase equilibrium studies were continued in the MgO-BeO, CeO_2-BeO, ZrO_2-BeO, and BeO-CaO systems. It was concluded that BeO and MgO do not form compounds or extensive solid solutions, although the possibility of very limited solubility of either constituent exists; the eutectic contains 67 ± 2 mole % BeO and the eutectic temperature is 1860 ± 20°C.

NUCLEAR TECHNOLOGY AND GENERAL SUPPORT

Fuel Element Catastrophe Studies. – Additional results of diffusion experiments on the release of fission products from reactor-grade PWR-UO_2

pellets in purified helium atmosphere at temperatures below melting indicated an increase in the slope of the release rate curve at temperatures above 1800°C. In 5 hr of heating at 1800°C, 3.7% of the rare gases were released, as well as 11.7% of the iodine, 21% of the tellurium, 3.2% of the cesium, 6.9% of the ruthenium, and 1% of the strontium. At 1900°C the values were 9.7% for rare gases, 16.6% for iodine, 48% for tellurium, 8.6% for cesium, 8.5% for ruthenium, and 2.3% for strontium. At 1980°C the values were 12.3% for rare gases, 41.6% for iodine, 75.8% for tellurium, 15% for cesium, 13.4% for ruthenium, 4.2% for strontium, 8.7% for barium, and 1.8% for zirconium. The amount of vaporized UO_2 was negligible.

The release of rare gases was shown to be quantitative from small specimens, no larger than 0.2 to 0.3 g of UO_2, mounted in BeO. Upon melting in helium, 90% of the iodine, tellurium, and cesium, 70% of the ruthenium, 0.2 to 3.9% of the strontium, and 2 to 7.3% of the barium are volatilized. By comparison, on melting in air, the results are slightly higher for iodine (95%), slightly lower for tellurium (80%) and cesium (40%), and much lower for strontium (0.1 to 0.3%) and barium (0.5 to 0.6%).

From vaporized uranium oxide collected during melting (about 10% in air), it was determined that the particles are composed of well-defined U_3O_8 crystals measuring 0.02 to 0.2 μ in diameter when the sample was melted in air, and that the particles are spheres of UO_2, 0.0075 to 0.03 μ, from samples melted in helium. The amount of U_3O_8 vaporized in air is at least an order of magnitude higher than the amount of UO_2 vaporized in helium.

Reactor Evaluation Studies. — The fuel value of plutonium in Dresden-type reactors was evaluated as a function of the fabrication cost associated with plutonium-containing fuel elements. If the fabrication cost for plutonium elements is no higher than for uranium elements, it appears economical to utilize the plutonium product as recycle material. Under these conditions the value of fissile plutonium is about \$11/g for the first recycle and about \$9/g for the second recycle. Increasing the fabrication cost of plutonium elements by \$60/kg decreased the fissile-plutonium value from about \$10/g to zero.

Calculations were also made for determining the optimum uranium enrichment with which to blend plutonium. It was assumed that various amounts of commercially pure plutonium were blended with uranium containing 0.71, 1, and 2% U^{235} respectively. With the fabrication cost the same as for uranium fuel elements, the value of fissile plutonium was \$14/g, independent of the initial U^{235} enrichment of the fuel. When the fabrication cost of plutonium elements was \$15/kg higher than for uranium elements, the fissile-plutonium value was \$13.4/g with natural or 1% U^{235} enrichment and \$13.0/g with 2% U^{235} enrichment.

Chemical Behavior of Thermoelectric Materials. — The refractive indices of potentially useful thermoelectric materials derived from UO_2 may be valuable indicators of the exact composition (O:U ratio) of the material, but are too high for the available immersion oils which measure only to 2.11. Sulfur-selenium glasses offer the possibility of extending this range from 2.11 to 2.70; to this end, wedges of such glasses have been prepared, and their refractive indices have been calibrated with the use

of an optical goniometer. The measurements were made with monochromatic light of 6500 A wavelength. The precision achieved by this method indicates that the refractive index of UO_2 can be determined by the Merwin technique with a precision of ±0.005 by immersing UO_2 crystallites in sulfur-selenium glasses of known refractive indices.

Solid State Reactions. – Preliminary results have been obtained for the diffusion of Co^{60}, Co^{56}, and Al^{26} in high-purity coarse-grained aluminum. The "infinitely thin layer" technique was used for Co^{60} and a thick foil technique for Co^{56} and Al^{26}. The diffusion coefficients (in cm^2/sec) were found to be: for Co^{60}, 4.6×10^{-8} at 639°C, 1.1×10^{-8} at 602°C, and 2.4×10^{-9} at 547°C; for Co^{56}, 5.2×10^{-8} at 640°C, 3.9×10^{-8} at 634°C, and 1.7×10^{-8} at 607°C; and for Al^{26}, 5.5×10^{-9} at 630°C.

Experiments to determine whether there is any relationship between the pressure of gases released during heat treatment and the degree of reaction of 50 wt % UO_2–Al compacts at 600°C show that the degree of reaction is a maximum at approximately 80 mm Hg and decreases at higher or lower pressures. The gas released in all cases was 96 to 99% hydrogen.

Fuel Element Development. – A program has been initiated on the brazing of ceramic materials to themselves and to metals. Approximately ten selected brazing alloys, which had previously been found to wet and flow on such difficult-to-braze materials as the refractory metals, were screened to determine their wetting characteristics on aluminum oxide. Three of the alloys showed promising flowability; they were then used successfully to braze titanium to aluminum oxide and zirconium to aluminum oxide. These brazing alloys were ternary compositions in the Ti-Zr-Be and Ti-Cu-Be systems.

Nondestructive Test Development. – Recent studies in the field of ultrasonic behavior in thin sections have been directed toward the detection and evaluation of nonbond areas in clad structures. A typical application of such studies would be to UO_2–stainless-steel dispersion-type flat plate elements clad with stainless steel, such as the fuel plate being developed at ORNL for use in the Fermi Fast Breeder Reactor. A through-transmission ultrasonic technique has proved to be the most feasible method for the detection of nonbond areas. One of the limitations of the technique is that normal variations in the dispersion core prevent the positive identification of a nonbond area less than 1/8 in. in diameter.

This nonbond problem is common to many fuel elements. A similar example is the one encountered on a flat plate element having an aluminum-clad uranium-aluminum core, which is being developed for the HFIR. In this particular case, there is a more homogeneous core, which allows detection of 1/16-in.-diam nonbond areas in spite of the difficulties associated with a variable core thickness.

Mechanical Properties Research. – A program is being conducted to establish the rupture life of Inconel at 1500°F under creep and low-frequency fatigue stresses. For rupture in 1000 hr, a simple relation has been found to exist between the fatigue stress amplitude, σ_a, and the mean creep stress, σ_m, which is of the form $\sigma_a = A - (A/B)\sigma_m$, where A is the fatigue stress to produce rupture in 1000 hr and B is the creep stress to produce rupture in 1000 hr.

Inconel has been strain-cycled under torsion at 1500°F to establish a strain absorption criterion for fracture. The results follow a previously established relationship for fracture ductility in creep under complex static stresses. Thus, these results add further evidence that the maximum principal stress criterion controls high-temperature time-dependent fracture even under dynamic stresses. It is, therefore, important that the stress state be considered in solving strain cycle design problems. Large errors will result if the principal strains are used instead of shear strains.

Materials Compatibility Studies. – The Materials Compatibility Laboratory is studying materials problems encountered in the containment of boiling alkali metals. These studies will be conducted on alkali metals which are attractive working fluids for reactor-turbine-generator power sources for space applications. Many of the systems evaluations have indicated that potassium is one of the most promising fluids, and studies to date have been concentrated on this metal.

Preliminary purification experiments indicate that the oxygen content of commercial potassium can be reduced to less than 30 ppm by hot-gettering with titanium sponge followed by cold-trapping at temperatures near the melting point of potassium.

Nine compatibility tests with refluxing potassium at boiler temperatures of 1500 and 1600°F have been conducted to date. The purpose of these tests is to provide screening information regarding the comparative corrosion resistance of Fe-, Ni-, Co-, and Cb-base alloys under various thermal conditions for several time periods. Weight-change and metallographic data for the test specimens indicate that Inconel (Ni-base) has less corrosive resistance than type 316 stainless steel (Fe-base) or Haynes alloy No. 25 (Co-base).

The most promising alloys are being tested in boiling potassium loop systems.

Fuel Cycle Technology. – Several 1-kg batches of water-dispersible thoria have been prepared by denitration of hydrated thorium nitrate with superheated steam. Temperatures of 390 to 450°C for about 1 hr gave oxides having 4.5 to 3.3 wt % volatile matter, which contains 4.6 to 2.6% H_2O and 1.4 to 0.8% N_2O_3. In tests of continuous steam-stripping of thorium nitrate solution in an agitated trough calciner, there was excessive entrainment of partially denitrated product. Batch denitration of solid $Th(NO_3)_4 \cdot 4.9H_2O$ in the same apparatus gave no entrainment from the reactor, but produced a product which contained 7 wt % volatile matter, whereas 3.3 to 4.5% is considered optimum. In the preparation of hydrosols of steam-denitrated thorium oxide, dispersion of particles was maximum in a 0.26 M nitrate solution when the solids content was 0.24 g/ml. For 5% UO_3–ThO_2 particles at a solids content of 1.3 g/ml, maximum dispersion was obtained in 0.44 M nitrate; for 3.9% UO_3–ThO_2 at 0.9 g/ml, it was obtained in 0.38 to 0.42 M nitrate. Dispersion of steam-denitrated materials was slow for about 50% of the solids, but the rate was increased by increasing the temperature and nitrate concentration and by at least an order of magnitude by vigorous stirring. During evaporation of sols to gels at about 80°C, nitrate concentrations above optimum led to small, glassy needle-pointed, sharp-edged particles which were not compacted well by vibration. Gel particles resulting from sols having poorly dispersed solids were large but porous.

Power Reactor Fuel Processing: Processes for Zirconium-Containing Fuel. — The rate of dissolution of samples of PWR seed 3 (34% UO_2–66% ZrO_2) in refluxing 3 to 12 M HF increased from 0.4 to 3.3 mg cm^{-2} min^{-1}. In other tests they were chlorinated with carbon tetrachloride vapor at a rate of 3 mg cm^{-2} min^{-1} at 595°C.

A modified Zirflex process compatible with stainless steel equipment was developed for recovering uranium from spent TRIGA reactor fuel (aluminum-clad 8% U–91% Zr–1% H). The cladding and the aluminum-samarium burnable poison disks are first dissolved in 2 M NaOH–1.78 M $NaNO_3$. The graphite moderator plugs are then disintegrated with a cold mixture of 90% 21 N HNO_3 and 10% 40 N H_2SO_4. The core is then dissolved in 5.4 M NH_4F–0.33 M NH_4NO_3–H_2O_2, and nitric acid and aluminum nitrate are added to produce a stable solvent extraction feed 0.0085 M in U, 0.25 M in Zr, 0.88 M in Al, 2.1 M in F, 1 M in HNO_3, and 0.7 M in NH_4. The entire process takes 14 hr. The decladding off-gas is ammonia and the core dissolution off-gas contains 10% H_2, 0.5% O_2, 2% N_2, and 87% NH_3.

Corrosion tests in which LCNA, Hastelloy F, and types 309SCb and 304L stainless steel were exposed to boiling 5.4 M NH_4F–0.33 M NH_4NO_3–0.003 M H_2O_2 in a flowing system showed maximum rates of 1.43, 2.16, 1.14, and 7.16 mils/month, respectively, for exposures of 144 hr. Type 304L stainless steel suffered extensive localized attack. The concentration of H_2O_2 varied between 0.0028 and 0.0032 M throughout the tests.

Power Reactor Fuel Processing: Processes for Beryllium- and Niobium-Containing Fuel. — Leaching of GCRE fuel pellets, 70% UO_2–30% BeO, for 65 hr in boiling 6 to 13 M HNO_3 recovered 99.9% of the uranium; approximately 50% of the BeO remained as an undissolved residue. In boiling 8 M HNO_3–0.2 M NaF the pellets dissolved completely in 30 to 40 hr. Pellets containing 66 to 70% UO_2, balance BeO, disintegrated to powder when heated in air at red heat. Niobium dissolved at a rate of 5.5 mg cm^{-2} min^{-1} in 30 M KOH at 325°C.

Power Reactor Fuel Processing: Solvent Extraction. — Solutions resulting from the leaching of uranium-graphite fuel with 90% HNO_3 may be adjusted to solvent extraction feed conditions by evaporating to a boiling point of about 135°C and then diluting with water. The capacity of a plant using the Acid Thorex flowsheet may be increased with no apparent change in decontamination by increasing the concentration of thorium in the feed and adjusting the amount of solvent used. Extraction of uranium from an acid-deficient feed with an Acid Thorex flowsheet resulted in decontamination factors from ruthenium, zirconium, and rare earths of 23,000, 4400, and 1.3×10^6 respectively. Solvent extraction with tributyl phosphate did not remove Nb^{95} from Zr^{95}. A solution of Zr^{95} tracer containing less than 5% Nb^{95} was prepared by treatment with unfired Vycor glass. The adsorption of Nb^{95} on Vycor was 90% complete in 5 min.

Tests to optimize the performance of the Immi mixer-settlers in cell 1 of Building 4507 showed that stage efficiencies of 92% are obtainable for uranium stripping with modified impellers run at 1600 rpm. Under these conditions the capacity of the Immi equipment for uranium recovery with the Purex flowsheet was 37.2 kg of uranium per day. The optimum extraction-scrub stage efficiency, 90 to 92%, was at an impeller speed of 950 to 1100 rpm. The equivalent capacity of the Immi is 26 kg of uranium per day. The total volumetric throughput of the Immi for extraction-scrubbing is 280 ml/min and for uranium stripping, 550 ml/min.

Power Reactor Fuel Processing: Criticality and Corrosion Studies. — The evaluation of boron glass Raschig rings for use as a fixed nuclear poison in fuel processing was continued. A titania coating on 1.5-in.-diam borosilicate rings containing 5.81% boron did not prevent chemical attack during 650 hr exposure to 2 and 6 N HNO_3 at 65°C. Visual observation showed partial loss of the coating, generalized crazing of the glass surface, and a few internal cracks.

In continuing corrosion tests the presence of 0.2 M borate in refluxing Thorex solvent extraction feed solution containing 0.04 M fluoride did not inhibit the vapor-phase corrosion of titanium. Titanium-45A was corroded at maximum rates of 0.04 and 2.01 mils/month in boiling initial Purex waste solution and in boiling 15 M HNO_3–1.0 M H_2SO_4, respectively, in exposures of 1000 and 1145 hr. LCNA and Hastelloy F specimens exposed in the vapor above boiling 6 M HNO_3 were corroded at the same rate as those above 6 M HNO_3–0.05 M H_2SO_4. These rates increased from 0.62 and 0.80 mil/month at 168 hr to 1.89 and 2.35 mils/month at 672 hr exposure respectively.

In tests at 35°C on possible materials of construction for the proposed multipurpose centrifuge, titanium-45A, Carpenter 20SCb, LCNA, Hastelloy C, and Hastelloy F were corroded at maximum rates of 0.01, 0.05, 0.03, 0.42, and 0.04 mil/month, respectively, in 336 hr exposure in Thorex solution. Corresponding rates in UO_2 core solution (10 M HNO_3–0.01 M Zr^{4+}–0.05 M NaF) were 11.1, 0.53, 0.38, 5.2, and 0.50 mils/month respectively.

Power Reactor Fuel Processing: Processes for Graphite-Containing Fuel. — In preliminary engineering-scale tests on the disintegration of nuclear-grade AGOT graphite with 90% HNO_3, 2-1/4-in. cubes of uranium-free graphite exposed to boiling dissolvent for 48 hr and to dissolvent at room temperature for an additional 48 hr were only partially disintegrated. All the graphite swelled, but some fragile chunks, 1 to 2 in. in diameter, remained. Slabs of 3% U–graphite were successfully disintegrated by 90% HNO_3 at 60 to 70°C in less than 1 hr. A settled bed of slurry from the latter test was readily transferred by vacuum through a 3/4-in.-diam tube to a filter tube containing 4 in. of 30-mesh Ottawa sand. The filtrate was completely clarified.

In laboratory tests the behavior of fission products in the 90% nitric acid process is being determined with slightly irradiated specimens (about 0.001% burnup). When specimens containing 3 and 12% uranium were leached twice for 4 hr at 25°C, both graphite residues retained 38–46, 28–80, 46–63, and about 75%, respectively, of the Zr^{95}, Cs^{137}, Ru^{106}, and Ce^{144}; the uranium losses to the two residues were 1.2 and 0.13% respectively.

Hydrolysis of UC by water at 80°C yielded principally a finely divided, insoluble uranium compound and 92 ml of gas per gram of UC. The off-gas contained 86% methane, 11% hydrogen, small amounts of ethane and propane, and a trace of ethylene. Hydrolysis of UC specimens containing about 20% UC_2 resulted in the evolution of 77 ml of gas per gram of sample. The off-gas from this sample contained 75% methane, 14% hydrogen, and 8% ethane. The off-gas from the high-purity UC contained 3.8 millimoles of carbon per gram, but only 3.4 millimoles/g was found in the UC-UC_2 mixture even though it contained more carbon per gram. Specimens containing UC_2 yielded some wax. In both experiments, carbon that was not accounted for was probably present as hexane and higher hydrocarbons, which were not detected by the analytical methods used.

Power Reactor Fuel Processing: Mechanical Processing. — Containment changes for the SRE decladding facility in Building 3026 are now 98% complete.

Initial tests to determine the feasibility of using a spiral inclined rotary drum as a feeder for the leacher indicate some back-mixing of solids when the feeder is operated at an angle of 20 or 30° from the horizontal. The rate of discharge of four 3-liter batches of solid steel rods (1/2 in. in diameter and 1 in. long) varied from 90 to 98% per revolution at 25° and 70 to 90% per revolution at 30°.

A material balance was calculated for the leaching of UO_2 from 1-in.-long pieces of stainless-steel-clad UO_2 with nitric acid in a ten-stage countercurrent inclined drum rotary leacher (four leaching, six washing stages). In steady-state operation of a shear-and-leach plant operating on typical tubular fuel bundles (mark 1 prototype), minimizing water and acid usage and using air for oxidation of NO to NO_2 to increase acid recovery, the main product overflow averaged 2 M $UO_2(NO_3)_2$–2 M HNO_3 (at 25°C) before mixing with wash water streams.

Fluoride Volatility Processing. — The first complete process flowsheet demonstration run was made with two enriched uranium fuel elements. The utilization of HF was 35%, higher than in previous runs in which dummy Zircaloy-2 elements were used. Operation of the new fluorinator and sodium fluoride absorbers was satisfactory. The UF_6 product was collected in a small product cylinder, and the percentage recovery will not be known accurately until this is analyzed. The facility for product cylinder sampling is under construction.

Data on the removal of solids from the hydrofluorinator off-gas in the eight dissolutions made to date indicate that a copper mesh cartridge is the most effective method of separation. Two cartridges in series, with improved instrumentation, should provide data for design of the unit to be used in radioactive runs.

Measurement of the INOR-8 hydrofluorinator with the Vidigage ultrasonic thickness gage after 204 hr exposure to anhydrous HF and NaF-LiF fused salt at 500 to 650°C during dissolution of Zircaloy-2 elements showed the maximum corrosion to be in the parts exposed to the vapor phase. Average corrosion rates were 0.045, 0.021, and 0.003 mil/hr, respectively, for exposure to the vapor, at the interface, and to the liquid.

PHYSICAL RESEARCH PROGRAM

Reactor Operations. — The ORR operated well during the cycle which ended January 15. Only one unscheduled fuel loading was required when water leaked through a control-rod drive tube causing a magnet to short and fail. A considerable effort is being made for improving the control-rod system which caused several days of extra shutdown time at the end of the last cycle.

Operation at the Graphite Reactor and the LITR was uneventful except for two minor radiation incidents from experiments at the LITR.

Waste Disposal. — Design is complete on the second seepage trench for intermediate-level waste, which, when finished, will provide capacity for all ILW and will permit the operation of the open waste pits to be terminated. The pits will be filled and sealed off as soon as possible.

Monitoring instruments in the creek system which will enable better estimation of the sources of waste are almost complete.

Gaseous waste monitoring has been successful in identifying several small sources of radioactivity. An improved design of gas-monitoring instrumentation, prepared by Tracerlab under contract with ORNL, is being studied.

Hot-Cell Operations. — Operation is continuing in cells 4 and 6 of the 3025 Physical Examination Hot Cells and has begun in cell 3. Equipment is being prepared for the first large job in cells 1, 2, and 3. This job will consist in the examination of an SM-1 fuel element, which will be measured, sectioned, and given various metallographic tests.

The south hot cell at the ORR was used about 50% of the time and the north cell about 10% of the time during the last quarter.

In the Building 3026D Segmenting Cells, installation of fuel dejacketing equipment in cell A continued. The B2 cell was used about 40% of the time. To date there has not been a sufficient backlog of work to require a second-shift operation of any of the cells.

PHYSICS AND MATHEMATICS RESEARCH

Neutron Diffraction. — Neutron diffraction investigations have been performed on a group of rare-earth nitrides and antimonides with the rock-salt structure to determine whether magnetic ordering exists in these compounds. HoN, TbN, and ErN become ferromagnetic at about 18, 43, and 6°K, respectively, and no magnetic ordering was observed in TmN at temperatures down to 1.3°K. On the other hand, HoSb and TbSb become antiferromagnetic at low temperatures, with magnetic structures in which the moments are arranged in ferromagnetic sheets normal to the cube diagonal and adjacent sheets are oriented antiparallel. The paramagnetic scattering from all these compounds agrees satisfactorily with moment values calculated for the free trivalent rare-earth ions, but the moment values in the ordered magnetic structures are lower than the calculated values. Recent calculations by Trammell [J. Appl. Phys. 31, 362S (1960)]

indicate that these low moment values and the specific orientation of the ordered moments are the result of crystalline field interactions. Diffraction patterns from the ferromagnetic rare-earth nitrides (particularly HoN and ErN) show considerable ferromagnetic short-range-order scattering which has characteristics that are different from those associated with critical magnetic scattering.

Theoretical Physics. – In the Brueckner theory the first-order contribution of the reaction matrix t to the energy per particle in an infinite medium of fermions is proportional to $\int_F d^3K\, d^3p\, (Kp|t|Kp)$, where $\vec{K}$ and $\vec{p}$ are the center-of-mass and relative momenta of a pair, respectively, and integration is over the Fermi sphere, F. The occurrence of reaction matrix singularities apparently implies the failure of the Brueckner theory, and, as L. N. Cooper has shown, singularities may occur for an arbitrarily weak interaction. This difficulty may be removed by obtaining the theory of the infinite medium from that of a finite system of volume V by taking the limit $V \rightarrow \infty$ with average density held constant. For a finite system the energy shift $\Delta E^{(V)}_{\vec{K}\vec{p}}$ for a pair differs from the corresponding reaction matrix element by a normalization factor, and unlike the latter does not have singularities. The energy per particle in the infinite medium is then proportional to

$$\lim_{V\rightarrow\infty} \sum_{K,p} \Delta E^{(V)}_{\vec{K}\vec{p}} = P \int d^3K\, d^3p\, (Kp|t|Kp) \; .$$

Because of the principal value (P) the singularities of t do not invalidate the perturbation solution. In place of the nonexistent continuum solution of the Bethe-Goldstone equation at a singularity of t, there exists a positive-energy bound-state solution. Its level shift is zero, corresponding to the change of sign of $(Kp|t|Kp)$ in going through the singularity. The terminology "bound state" refers to the fact that the wave function vanishes for large interparticle separation. The existence of true bound states imbedded in the continuum is a consequence solely of the Pauli principle. The positive-energy bound states must lie between zero and the Fermi level and therefore disappear entirely as the density goes to zero. For a combination of sufficiently strong attraction, low density, and low center-of-mass momentum, negative-energy pair states exist. Unlike positive-energy bound states, negative-energy pair states are not in correspondence with unperturbed states. Therefore, to first order in t (or in the independent pair model) a state lying lower than the lowest state of perturbation theory is obtained by incorporating negative-energy bound states. (Since their eigenvalues are not inversely proportional to the volume, a nonnegligible addition to the binding energy is obtained if there exists a negative-energy pair state for each K in a finite interval.) The situation is obscured for physical applications by the fact that, as the attraction is increased, the saturation density also increases (up to a point). However, the existence of diatomic molecules indicates the relevance of negative-energy pair states, and it is hoped that by their inclusion the independent pair model can

be applied to molecular physics, where the interaction is relatively much stronger than that between nucleons.

Low-Temperature, Nuclear and Solid State Physics. – A study by the Mössbauer method of the hyperfine splitting of Au^{197} alloyed in iron, cobalt, and nickel has been made. Au^{197} has a $3/2^+$ ground state and a $1/2^+$ first excited state at 77 kev. It is found that the effective magnetic field at the gold nucleus is 280,000 gauss in the iron alloy, 215,000 gauss in the cobalt alloy, and close to zero in the nickel. An attempt is being made to correlate these fields with ideas about the polarization of the conduction electrons.

In each of these alloys there is a very large "chemical shift." This chemical shift is related to the product

$$[\psi_s^2(0, \text{alloy}) - \psi_s^2(0, \text{source})]\delta r/r ,$$

where the difference between the ψ's gives the difference between the amount of electron charge within the nucleus between alloy and source, and the $\delta r/r$ are then related to the difference in nuclear volume between excited and ground states. Thus the chemical shift measures a kind of nuclear thermal expansion coefficient. The fact that the chemical shift in this system is much larger than in any other system thus far studied may be related to the fact that gold is an odd-proton nucleus.

In the cobalt and iron alloys the two Mössbauer lines differ in width by approximately 30%. This may be due to a quadrupole splitting of the ground-state levels. If so, it indicates a large "covalent" bonding of the gold to a distortion of its surroundings. This is unexpected, in that metallic bonding is not usually considered to be appreciably covalent in character.

High-Voltage Experimental Program: Mössbauer Effect for Ni^{61} with Applied Magnetic Fields. – The Mössbauer effect with Ni^{61} was observed in the presence of applied magnetic fields. The Ni^{61} nuclei, for both source and absorber, occupied normal lattice sites in the nickel crystal lattice. The application of external fields served to polarize the internal fields, which are responsible for the hyperfine splitting in the source and the absorber. Consequently, the nuclei involved in the Mössbauer emission and absorption acted respectively as polarizer and analyzer of the individual gamma transitions. A measurement of the Mössbauer spectrum M(v) with applied magnetic fields yields certain information relating to the nuclear magnetic moments μ_e and μ_g and the internal magnetic field H. From this experiment the ratio μ_e/μ_g was found to be -0.47 ± 0.08 and the product $(\mu_g \cdot H)/\hbar$ was found to be 26.0 ± 2.6 Mc/sec. The field H and the magnetization J were found to be opposite in direction, just as they are in the case of Fe^{57}. Special cases of a general expression for the Mössbauer spectrum of Ni^{61} were derived for several geometries which are particularly suitable for experimental investigation.

High-Voltage Experimental Program: Fast Time-of-Flight Activity. — Attempts to observe prompt gamma rays from 30-kev neutron capture reactions proved to be successful. Neutrons (kinematically collimated to ±10° in the forward angle) are supplied by ~10^{-8}-sec proton pulses from the ORNL 3-Mev pulsed accelerator operating at 1 ± 0.1 kev above the 1881.1-kev $Li^7(p,n)$ threshold. Capture samples are located at 0°, 50 cm from the target. The detector is a large (9 × 6 in.) NaI crystal housed in a shield of lead and $(CH_2)_n$-Li^6F. Capture events are recorded in the 2048-channel analyzer, dynamically gated in time corresponding to regions of interest in the time spectrum. Thus one section of analyzer memory records background while the other simultaneously records gamma-ray counts at the precise moment of neutron capture by the sample. Preliminary spectra have been measured for capture gamma rays from samples of Ag, Au, Hg, Dy, Fe, and Ni.

CHEMISTRY RESEARCH

Solvent Extraction Research. — Two promising reagents for stripping extracted plutonium without introducing sulfate, metal ions, or gas-forming contaminants into the aqueous plutonium product were found: di-*tert*-butylhydroquinone (stripping by reduction) and aqueous acetic acid (stripping by displacement and competitive complexing). The dibutylhydroquinone is nearly insoluble in the aqueous solution but soluble to at least 0.03 *M* in Amsco 125-82 containing 0.3 *M* tertiary amine and 10% tridecanol, and at that concentration gave $S_o^a \approx 100$. Stripping with acetic acid was similar to that with sulfuric acid, the S_o^a increasing from ~10 at 0.3 *N* to >50 at 3 *N*.

According to a tentative flowsheet for separating transplutonic elements from fission product rare earths, the tributyl phosphate extract of the mixed elements from neutral nitrate solution is scrubbed with 12 *N* LiCl–0.1 *N* HCl to remove nitrate. It is stripped with 7 *N* LiCl, and the salt concentration of the strip solution is adjusted to 8 *N* LiCl–2 *N* $AlCl_3$. This is fed to a multistage partitioning system, with 0.5 *M* tertiary amine in a modified aliphatic hydrocarbon as the extractant and 8 *N* LiCl–2 *N* $AlCl_3$ as the scrub. In continuous operation with no external reflux, the americium/europium separation factor was 10 for each stage, with curium extraction slightly less than that for americium.

The alkaline permanganate method for recovering 1 *M* TBP in Amsco 125-82 that has been exposed to radiation in the presence of nitric acid and scrubbed with sodium carbonate to remove low-molecular-weight acids required only 5% as much reagent as the alkaline aluminum method. The alumina and the manganese dioxide formed during treatment showed similar sorption for the remaining degradation products. Impurities causing 50 to 60% of the Zr-Nb extraction of the degraded solvent were easily sorbed; removal of another fraction, causing 20 to 25% of the extraction, required further relatively large additions of solid, but even at the highest solids levels used, the solvent retained 20 to 25% of its Zr-Nb extraction power.

Ion Exchange Studies. — The rates of sulfate and chloride self-diffusion from different size fractions of Dowex 21K into dilute Na_2SO_4 and

NaCl solutions were measured as a function of the solution flow rate through the resin. The solutions were dilute enough (0.0005 M) that the resistance to diffusion was largely in the liquid film surrounding the resin. These results should give values for the effective film thicknesses which will be of use in the treatment of uranium ion exchange data. Preliminary analyses of the data indicated that the ratio of the film-to-particle diffusion for sulfate self-diffusion from 1200-μ Dowex 21K, $rD_{film}C_{soln}/\delta D_{resin}C_{resin}$, is approximately 0.020.

Mechanisms of Separations Processes: Thermodynamics of Extraction with TBP. — As a basis for systematizing data on the extraction of uranium and other cations from nitric acid solution by TBP-hydrocarbon diluent, extraction of nitric acid by TBP-Amsco 125-82 is being studied for aqueous HNO_3 concentrations of 0.1 to 8 M and TBP concentrations in the diluent of 100 and 30%. Based on the model

$$H^+_{aq} + NO_3^-{}_{aq} + TBP_{org} \rightleftharpoons TBP\cdot HNO_{3org} \quad ,$$

the activity coefficient quotient is accurately described by

$$\log \frac{K_1 y_{TBP,org}}{y_{TBP\cdot HNO_3,org}} = \log \frac{(HNO_3)_{org}}{[HNO_3]_{aq}\ (TBP)_{org}} = a + b\ \frac{(HNO_3)_{org}}{(TBP_s)_{org}} \quad ,$$

where y is a molar activity coefficient, square brackets refer to activities, parentheses refer to concentrations, TBP_s refers to total TBP in the organic phase, and K_1 is the extraction equilibrium constant. For 100% TBP and 30% TBP in Amsco 125-82, the constant a is 0.176 and −0.149 respectively; b is −1.59 and −1.047. Since the activity coefficient of $TBP\cdot HNO_3$ in the organic phase can be defined as 1 at infinite dilution, the values of parameter a correspond to $\log K_1 y_{TBP,org}$. The decrease in a as TBP is diluted with hydrocarbon corresponds to a decrease in the activity coefficient of TBP in water-saturated TBP-Amsco 125-82 solutions.

Mechanisms of Separations Processes: Foam Separation. — In foam separation, being studied under subcontract with Radiation Applications, Inc., as a means of separating elements from solution and from each other, a gas, such as nitrogen, is bubbled through a feed solution to which a surface active agent has been added. If this surfactant forms a complex with the cations in solution, for example, cesium, calcium, and strontium, the bubbles formed are enriched in these elements, while the solutions are depleted.

Fundamental studies have shown that, with the organic sodium sulfonate RWA-100 as the added surfactant, calcium, strontium, and sodium are distributed between the bubbles and the solution phases according to the Gibbs and Langmuir equations:

$$\frac{\Gamma}{c} = -\frac{1}{nRT}\frac{d\gamma}{dc} = \frac{\Gamma'_m \alpha}{1 + \alpha c} \quad ,$$

(Gibbs) (Langmuir)

where Γ'_m is the monolayer surface concentration of the component being studied, Γ is the excess surface concentration, c is its bulk concentration, γ is the surface tension, R and α are constants, and T is the temperature. This correlation applies to data from a single-stage foam separation column and to surface tensions determined by surface radioactivity measurements as well as surface tensions measured with standard ring tensiometers.

Physical Chemistry of Molten Salts. — In order to obtain reliable data with which to test the generalized theoretical calculations for stepwise association constants (ORNL-2931, p 47), a method was developed and applied to the evaluation of these constants at infinite dilution for the association of Ag^+ and Cl^- in molten KNO_3. With the concentrations expressed as moles per mole of KNO_3, the stepwise association constants for the formation of AgCl, $AgCl_2^-$, and Ag_2Cl^+ were found to be, respectively, 456 ± 8, 169 ± 20, and 70 ± 15 at 385°C.

From phase equilibrium studies of the systems $KF-ThF_4$ and $RbF-ThF_4$, new phase diagrams were constructed which are significantly more complex than those derived elsewhere. In this investigation one new $KF-ThF_4$ compound and four new $RbF-ThF_4$ compounds were characterized. Their optical properties and characteristic x-ray diffraction data were obtained.

The optical properties and x-ray data were obtained for the compound $3CrF_2 \cdot 2CrF_3$. The compound is biaxial negative, with $2V \approx 55°$, $N_\alpha = 1.518$, and $N_\gamma = 1.534$. The compound melts incongruently at 1000 ± 4°C to CrF_3 and liquid. No evidence of polymorphism in $3CrF_2 \cdot 2CrF_3$ has been observed.

High-Temperature Aqueous Solution Chemistry: Hydrogen Solubility. — The solubilities of H_2 in H_2O and D_2 in D_2O were determined at 10° intervals from room temperature to 250°C. The data are believed accurate to ±1%. The H_2-H_2O results are in essential agreement with those of Wiebe and Gaddy [J. Am. Chem. Soc. 56, 76 (1934)] below 100°C and with those of Pray et al. [Ind. Eng. Chem. 44, 1146 (1952)] above 100°C. The D_2-D_2O results indicate solubilities which are several per cent lower than those reported in BMI-1067 for temperatures of 162, 205, and 260°C.

High-Temperature Aqueous Solution Chemistry: Isopiestic Studies. — Modifications have been made in the apparatus for the isopiestic study of aqueous solutions of electrolytes at elevated temperatures. The indirect electronic null-point device for sensing the position of the balance was replaced with an optical system by which a narrow, collimated light beam is reflected from a mirror on the balance through a small optical port in the side of the pressure vessel. Direct observation of the balance gave an improvement in weighing accuracy and greatly decreased the time required for the weighing process. With the improved apparatus the previously reported studies at 100°C with representative electrolytes (LiCl, NaCl, KCl, CsCl, Na_2SO_4, $BaCl_2$, $MgSO_4$, UO_2SO_4) (ORNL-2931, p 133; J. Chem. Soc. 1960, 2730) were extended to 110, 120, 130, and 142°C. Comparision of the results with the 100°C data and with literature values for 25°C revealed no inconsistencies attributable to the modification of the apparatus. The isopiestic ratios of the 1-1 electrolytes vs NaCl were unchanged from 25 to 142°C except for LiCl; LiCl, which resembles HCl (a

commonly used standard), showed over 12% change in isopiestic ratio from 25 to 142°C, suggesting that caution should be used in interpretations of behavior at elevated temperatures based on HCl standards. The 1-2, 2-2, and 2-1 electrolytes showed definite indications of linear relationships between isopiestic ratio and temperature, with the slopes being functions of the charge type.

Physical Chemistry of Ion Exchangers. — Measurements of the excess free energies and heats of dilution as a function of concentration have been completed for a number of quaternary ammonium halide salts in an attempt to understand the origins of the ionic selectivities and heats of exchange exhibited by strong-base organic anion exchangers.

Osmotic coefficients of tetramethyl, benzyltrimethyl, and benzyldimethylethanol ammonium chloride and bromide in aqueous solutions were determined by using the gravimetric isopiestic vapor pressure method. In addition, chloride-bromide salt mixtures were measured. Ion binding, as reflected by lowered osmotic coefficients, appeared to increase (1) as the size of the quaternary ammonium ion increased and (2) in going from the pure chloride to the bromide salt. It could be inferred also that important contributions to the selectivity coefficients of anion exchangers for halide ions must come from the structurally bound quaternary ammonium exchange groups in these materials.

Dilution of the aqueous solutions of the chloride, bromide, and iodide salts of the benzyldimethylethanol ammonium ion (Dowex 2 analog) was accompanied by the absorption of heat. The fluoride salt, however, evolved heat as is typical for the dilution of salt solutions with hydrated ions. The standard heats of exchange observed previously for variously cross-linked Dowex 2 anion exchangers were compared with the differences in the apparent molal heats of dilution of solutions of the corresponding quaternary ammonium halides over the appropriate concentration range. For the bromide-chloride and the bromide-fluoride exchange systems the heat of dilution differences were only 20% of the exchange heat shown by a nominal 0.5% DVB exchanger. With 10 and 24% DVB exchangers, however, these differences amounted to approximately 90% of the observed heats of exchange. Unfortunately, the solubility of the quaternary ammonium iodide was not sufficiently high to permit comparisons with the iodide-bromide ion heat of exchange for any resin.

Thermochemistry and Calorimetry. — In two determinations of the heat of fusion of Li_3ThF_7 with the Bunsen ice calorimeter, values of 14,300 and 13,800 cal/mole were obtained. Premelting was observed 13° below the melting point of 573°C, corresponding to an apparent impurity content near 3 mole %, far larger than expected. Additional measurements are in progress to determine whether this phenomenon is associated with the Li_3ThF_7 itself and, also, to determine the source of the relatively large discrepancy.

Chemical Separation of Isotopes. — Construction of the water distillation cascade of the O^{17} pilot plant was completed, and shakedown operations are in progress. All columns in the thermal diffusion cascade were assembled and mounted. Completion of this cascade is expected next month.

Chemical Applications of Nuclear Explosions. — Tritium exchange from HTO to H_2 over $CaSO_4$ was independent of the surface area of $CaSO_4$ for residence times of 5.4 and 45 sec under diffusion-controlled conditions. The apparent activation energy for the reduction of $CaSO_4$ by H_2 was 39 kcal/mole.

Measurement of the temperature of a 97% Ar–3% H_2 plasma in a plasma jet by Stark broadening of the H_β line showed that the temperature increased from 10,500 to 12,500°K when the power density was increased from 86.5 to 128 w/std ft^3.

METALLURGY AND MATERIALS RESEARCH

Structure of Metals. — The deformation stacking fault probability in cerium deformed at room temperature was found to be about 0.030; the value for thorium was 0.020 (one fault for every 30 and 50 {111} planes respectively). The high faulting probability in cerium is due to the deformation temperature being close to the cubic-hexagonal phase transformation temperature. Thorium did not exhibit a behavior similar to that of nickel, as would have been expected on the basis of Seeger's theory.

CONTROLLED THERMONUCLEAR RESEARCH

Cross-Section Studies. — Modulation frequencies of 120 cps arising from the ion source power supplies, although small at the source, have affected the beam intensity by 50% at the detection site. This modulation subsequently prevented the detection amplifiers from responding to the modulation frequency (50 cps) of the target electrons. This modulation has been reduced to 0.1% by better regulation of the power supplies. Only a solution of the problem of short-term drifts of the beam (over periods of a few seconds) remains in order to realize the regulation necessary for crossed-beam measurements.

Assembly of the proton source for the $H_2^+ + H^+$ crossed-beam measurements in the DCX is nearing completion, and preliminary tests have given a proton beam of 20 ma at 60 kev in a spot 3/4 in. in diameter.

DCX-1 Facility. — After some initial difficulties the new 600-kv isolation transformer stack now seems to be operating satisfactorily.

Tests of the High Voltage Engineering Corporation ion source in conjunction with the DCX-1 accelerator tube were continued. An arc discharge between the extractor and the einzel lens resulted in a leak in the cooling oil supply to the lens. The source and magnetic analysis assembly and the accelerator tube were removed from the facility for cleaning. The lens was repaired and the source reassembled with some modifications. One porcelain section of the accelerator tube cracked after reassembly, and the tube is now being repaired.

Two new gas arc anodes are being fabricated. In these the arc strikes water-cooled stationary tubes, arranged in one case for vortex cooling and in the other for swirl cooling.

DCX-EP-A. — The demineralized water system problems in Building 9201-2 were solved temporarily, and the DCX-EP-A was put into operation on January 26.

The following characteristics of EP-A are noteworthy: (1) There are ten power supplies, with ratings from 2.5 to 500 kv and from 200 to 3500 amp. There are 56 welders, which can be easily connected in any series-parallel system desired. (2) The vacuum system consists of two 20-in. diffusion pumps upon the liner and three 20-in. diffusion pumps connected to the outer tank system. (3) The magnetic system consists of two mirror coils and four outboard coils. The facility is so constructed that any or all of the coils may be energized separately, thus making the usable magnetic length capable of being wound from 30 to 200 in. The ratio of fields between mirror and outboard coils can also be easily adjusted. (4) A 150-kv neutralized accelerator tube is to be completed during the first part of February.

The EP-A was made as adaptable as possible, and the various supplies and pumping arrangements will contribute greatly to the planned program.

Ion Source Development. — The modifications of the 600-kv beam test facility should be completed within the next two or three weeks. At this time, studies of beam trajectories, injector shields, and targets will be continued.

A solenoid lens has been built for the 100-kv ion source test facility which should make it possible to analyze ion currents up to 500 ma. Utilization of this facility has been delayed until the 7000-amp rectifiers for the solenoid are installed. This installation should be completed within the next two weeks.

Theory. — The stability of a rotating infinite cylindrical shell of plasma has been analyzed. This model, which more closely resembles the DCX than previous ones, has qualitatively the same dispersion relation as before. Instability is predicted whenever the electron plasma frequency exceeds the ion cyclotron frequency. The theory also predicts that the unstable oscillations should have frequencies which are integral multiples of the ion cyclotron frequency. Nonlinear effects will limit the growth of these oscillations to rather modest values, however.

The Fokker-Planck coefficients for the radiation oscillators have been derived for the case of an infinite uniform plasma with no external fields. The numerical Fokker-Planck code has been modified and is now performing in a more satisfactory manner. Further tests are being conducted on it at present.

BIOLOGY AND MEDICINE PROGRAM

RADIATION EFFECTS ON BIOLOGICAL SYSTEMS

Genetic and Cytogenetic Effect of Radiations. — It has been found that the chromosome exchanges induced by x rays in *Tradescantia* microspores are not distributed randomly within the population of cells scored. The discrepancy can be accounted for on the basis that there are only four

sites within the nucleus where the broken strands are within the distance over which exchanges can occur. Chromatid exchanges that occur after the chromosome has doubled, however, are distributed at random in these same cells; that is, they are distributed according to the Poisson formula. In Vicia root tip cells, which have an average of only two such sites for chromosome exchanges, the chromatid exchanges, unlike those in Tradescantia, do not fit the Poisson distribution. The number of sites doubles after chromosome duplication, so that now there are four sites at which exchanges can occur, and the aberrations are distributed accordingly. It seems probable that in Tradescantia the number of sites doubles from four to eight after chromosome duplication, and that eight sites are enough to yield a fit to the Poisson distribution.

Cell Growth and Reproduction. - It has been demonstrated in embryos of the wasp Habrobracon that most of the deaths from radiation can be attributed to inhibition of mitosis. Radiation-induced damage in embryos of the fruit fly Drosophila melanogaster closely resembles that in Habrobracon. Most of the deaths occur during the first few nuclear divisions in both species when the eggs or sperm are irradiated.

Unfertilized but otherwise normal Habrobracon eggs were compared with young embryos containing genetically contrived chromosome bridges and with embryos developing from eggs fertilized by irradiated sperm. The unfertilized eggs stopped development immediately after the process of meiosis, but the irradiated embryos and the embryos with genetically contrived bridges continued through two to five mitoses before death. It appears likely that a considerable portion of the mitotic inhibition from radiation may be due to chromosome bridge formation.

Pathology and Physiology. - Male RF mice exposed at ten weeks of age to whole-body 250-kvp x radiation were injected intravenously with Myleran, 20 mg/kg, before or after irradiation, as follows: group I, Myleran 15 days before 150 r; group II, Myleran 5 days before 150 r; group III, Myleran 5 days after 150 r; group IV, Myleran 15 days before 300 r; group V, Myleran 5 days before 300 r; group VI, Myleran 5 days after 300 r; group VII, vehicle 5 days after 150 r; group VIII, vehicle 5 days after 300 r; group IX, vehicle alone; group X, Myleran alone. The incidence of thymic lymphoma in the respective groups (about 75 mice per group) was: I, 28%; II, 26%; III, 22%; IV, 38%; V, 34%; VI, 15%; VII, 5%; VIII, 16%; IX, 2%; and X, 9%. The incidence of myeloid leukemia was: I, 5%; II, 19%; III, 12%; IV, 4%; V, 21%; VI, 25%; VII, 29%; VIII, 31%; IX, 4%; and X, 1%. Thus the induction of lymphomas was increased and the induction of myeloid leukemias was decreased by Myleran to an extent depending on the time of administration, although the reverse was anticipated because of the myelotoxic action of the drug.

Biophysics: Magnetic Resonance Studies. - Following previously reported work on naturally occurring electron spin resonances in lyophilized cells of Escherichia coli B/r, the induction of free radicals in these cells by radiation has been studied. Preliminary experiments indicate that magnetic centers produced by x rays decay more rapidly in the presence of oxygen than in a vacuum, in contrast with the case for the unirradiated lyophilized material. A broad pattern with an apparent double peak which is observed after irradiation in a vacuum is heightened and

narrowed by heating to 100°C for 20 min. This change in pattern is suggestive of a heat-induced transition from one primary induced radical species to another.

The effects of heat and exposure to ultraviolet radiation following x-ray treatment have been studied in a number of systems, including Tradescantia and maize pollen, deoxyribonucleic acid, thymine, and glycine. In all instances, treatment with either heat (up to 100°C) or ultraviolet has resulted in a marked decrease in the x-ray-induced electron spin resonance signal, even in cases where exposure to ultraviolet alone results in a free-radical signal. For glycine this decrease has been correlated with striking changes in the ultraviolet absorption spectra of irradiated single crystals.

Evidence for saturation of free-radical production in glutamic acid at high radiation doses has been observed.

Nuclear magnetic resonance spectra of S,2-aminoethylisothiourea (AET), S,3-aminopropylisothiourea (APT), and their derivatives are being studied. By means of this technique it has been possible to follow closely the hydrolysis of these compounds.

Biophysics: Ultraviolet Studies. — The action spectrum for photoprotection in the bacterium Pseudomonas aeruginosa has been determined (four different wavelengths used). It is not appreciably different from the more complete spectrum done with Escherichia coli B. Apparently both organisms, although from different orders of bacteria, utilize the same chromophore for photoprotection. The survival curves at 2537 A were found to be the same in air and in nitrogen both for Escherichia coil B and for B/r.

Single cells of Amoeba proteus in a mineral medium have been irradiated at 2537 A and photoreactivated at 3000 to 4000 A. Division delay has been determined as a function of the dose of 2537-A radiation, and high photoreactivation has been found at several dose levels. These results are in general agreement with reports in the literature.

Cells of Escherichia coli C infected with the virus ΦX174, which contains single-stranded deoxyribonucleic acid (DNA), were irradiated with monochromatic ultraviolet light at different times after infection, and the ability of the virus-cell complex to make one or more virus particles was observed. The action spectrum for the inactivation of 45-sec complexes is similar in shape to that of the free virus. These data, plus previous results, indicate that right after infection the DNA is single-stranded, at 3 min it is double-stranded, and replicated DNA appears at 6 min.

Transforming principle DNA has been irradiated at 98°C by monochromatic ultraviolet light. At this temperature the DNA is single-stranded. The action spectrum for inactivation of the single-stranded material differs, as predicted, from the previously obtained spectrum for inactivation of double-stranded DNA.

Treatment of Tradescantia pollen with light in the wavelength region between 4200 and 6000 A before irradiation with ultraviolet light gave a marked degree of protection against chromosome aberrations, especially isochromatid breaks. When completely dry pollen was treated with light in this range (4200 to 6000 A) and was not exposed to ultraviolet, an unusual number of aberrations were produced.

Chemical Protection and Enzyme Catalysis. – The quantitative study of the response of various organ systems of the chemically protected mouse after total body irradiation has been extended to the spleen. A technique has been devised for making total cell counts of the spleen, and the response has been studied with both S,2-aminoethylisothiourea (AET) and S,3-aminopropyl-N'-methylisothiourea (APMT). Neither compound protected against the initial drop in cell count, although both compounds gave a rapid recovery of the cell population to normal values by day 20. In addition, APMT was found to yield hyperplastic spleens.

Blood platelet walls possess many unique characteristics in contrast with the walls of most other cells. Partial solubilization of platelet walls was achieved by extraction with alkali. Since the walls gave a positive hydroxamic acid test, the bonds broken in the extraction were probably ester bonds. The soluble portion was rapidly denatured, but a stable soluble material was obtained by an initial dinitrophenylation followed by alkaline extraction of the walls. The protein-like soluble material was ultracentrifugally heterogeneous and contained about 6% carbohydrate.

Equilibrium dialysis measurements of the binding of reduced diphosphopyridine nucleotide (DPNH) to purified mitochondrial malic dehydrogenase from beef heart have shown that 2 moles of DPNH are bound per 70,000 g of protein. This value is fairly close to the molecular weight of 65,000 that was previously obtained by sedimentation-diffusion data. In addition, it was found that one or more of the six half-cystine residues present in the molecule are "free sulfhydryl groups" essential for enzyme activity; 5×10^{-3} M iodoacetamide at pH 7.4 completely inhibited the enzyme. Diisopropyl fluorophosphate, an esterase inhibitor, had no effect on enzyme activity (at 1×10^{-4} M).

COMBATING RADIATION DETRIMENTAL EFFECTS

Radiation Protection: Living Cells. – The production of resistance to ionizing radiation in *Escherichia coli* by physiological means has been reported previously. Attempts were made to alter the survival of stationary-phase resistant cells by various short-term treatments. One promising approach involved a 3-hr incubation in sodium glutamate. Under these conditions, the cells raised the pH of the solution from 6.8 to 7.8 and the population became more sensitive. Repetition of this basic experiment under a variety of conditions showed that the incubation produced an increase in cell number whenever the population was made more sensitive. Analysis of the population with respect to cell size distribution established that an increase in total cell number was accompanied by the appearance of small cells. It appears, therefore, that although the cultures used in these experiments contained about 7×10^9 cells per milliliter, cell division was still a possibility and the population became sensitive. The kinetics of inactivation indicated that almost the entire population became more sensitive, even though the increase in cell number involved not more than 73% of the population.

Mammalian Radiation Recovery. — Histologic examination of the lymphatic system of bone marrow chimeras led to studies of the lymphatic system of normal mice exposed to a wide variety of antigenic materials. A common histologic reaction was discovered which appears to characterize the immune mechanism and its diseased states. Preliminary experiments suggest that the histologic reaction starts within 1 hr after rat bone marrow injection in the mouse.

Radiation Immunology. — Further results were obtained with Millipore filters employed as diffusion chambers in vivo. With chambers containing rabbit spleen cells and the test antigen (bovine serum albumin), the mitotic index of antibody-forming cells was found to be about four times as large as that of non-antibody-forming cells at the height of antibody globulin synthesis.

In the study of the potential primary antibody-forming capacity of nonimmunized spleen cells at varying intervals after culturing in isologous lethally irradiated mice, it was found that the characteristic gradual decrease in antibody-forming activity with time after culturing 12×10^6 spleen cells can be overcome by culturing 12×10^6 isologous bone marrow cells at the same time. It was also found that there was no decrease in activity with time when 200×10^6 isologous spleen cells were cultured in lethally irradiated mice.

Further analysis of data on the radiation inactivation of the antibody-forming capacity of spleen cells showed that the radiosensitive target, if spherical, has a diameter of about 0.4 μ.

The study of transplantation antigen has been hampered because the active component could not be solubilized. Recently, homologous hematopoietic transplantation antigen or antigens were obtained in the soluble fraction by the following procedure. Spleen cells (5×10^7 per milliliter) were treated as follows: (1) homogenization in Locke's solution (pH 7.4), (2) irradiation with ultrasonic waves for 20 min at ice temperature (Raytheon Sonicator R-22-3, 200 v), (3) enzyme treatment for 30 min at ice temperature (2.7 mg trypsin and 1 mg chymotrypsin per 8 ml of solution), and (4) centrifugation (90,000g for 45 min). Relative to nucleated cells, about 70% of the activity can be recovered in this manner.

TRAINING, EDUCATION, AND INFORMATION PROGRAM

International Reactor Programs at ORNL. — Fifty-two full-time students representing nine different countries have completed 12 weeks of school since the beginning of classes in November 1960. Twenty students are enrolled in Reactor Operations Supervision, eight in Reactor Hazards Evaluation, and 26 in other ORSORT programs. Tentative plans have been made for starting a fourth session of international programs in the fall of 1961.

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