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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

SEPTEMBER 1960

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Oak Ridge, Tennessee
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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

September 1960

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. – In further cleanup of contaminated areas, the penthouse floor was resurfaced by the Amercoat 74 treatment, which includes Fiberglas. The final installation of the dissolver and vessel off-gas line to the 3039 stack system increased the vacuum in process vessels from a few tenths of an inch to several inches of water. Building interim containment modifications were completed.

All process equipment was flushed with a solution of 2.0 M nitric acid containing 0.02 M fluoride and 0.04 M boron to determine if appreciable plutonium as polymer had been retained in the process vessels. Excluding the intercycle product evaporators, only 8 g of plutonium was found in the process equipment. Approximately 1100 g of plutonium was recovered from the intercycle evaporator equipment involved in a chemical explosion in November 1959, approximately 450 g of plutonium from the Turco solution drained from the evaporator prior to the explosion, and 650 g from the solution used to flush the steam-stripper and evaporator loop. Less than 2 g was recovered from the second intercycle evaporator.

REACTOR DEVELOPMENT PROGRAM

GAS-COOLED REACTOR PROJECT

EGCR Core Loading Studies. – The decision has been made to load the initial EGCR core in two zones of different enrichments. An annulus at the core boundary containing approximately one-half the fuel elements will be loaded with fuel of 2.4% enrichment. The remaining channels will be loaded with fuel of 2.2% enrichment. Although this step represents a complication in the design and presents problems in fuel management during operation, it was deemed necessary to provide additional power flattening. With this proposed zone loading, the reference reactor configuration, that is, experimental assemblies with carbon-to-uranium ratios of 200 in the central loops and the central control rod inserted, will have a radial peak-to-average ratio of 1.24, as compared with a value of 1.35 for a uniform core loading. This provides the essential margin below the maximum allowable

value of 1.35 to accommodate the greater fission cross sections of fresh fuel when refueling operations are initiated.

Of the several methods which are being considered for refueling the reactor, the one which appears to offer most promise consists in loading fresh fuel (~ 2.6% enrichment) into channels in the outer zone when the initial elements have reached (in the equilibrium cycle) an exposure of about 5000 Mwd/metric ton. The exposed fuel would then be inverted and shifted to a channel in the central zone, and the fuel from the central channel would be discharged to the pool. This fuel cycle is being compared with one in which the two zones are refueled with elements of different enrichments.

Hot-Channel Factors for EGCR Fuel Elements. – The hot-channel factors for the EGCR fuel elements have been brought up to date, based on the latest flux data and bank operation of the control rods to a maximum insertion depth of 62 in. The probability that the surface temperature of an element of the cluster will exceed the nominal maximum surface temperature of 1466°F by any given amount is given below, based on normal operating conditions:

Temperature Increase (°F)	Probability	Temperature Increase (°F)	Probability
0	0.5000	100	0.0209
10	0.4200	110	0.0125
20	0.3420	120	0.0074
30	0.2700	130	0.00405
40	0.2070	140	0.00219
50	0.1520	150	0.00111
60	0.1115	160	0.00056
70	0.0770	170	0.000275
80	0.0520	180	0.000124
90	0.0335	190	0.0000

These values do not allow for the effects of empty fuel channels or the increased reactivity resulting from a nonequilibrium core loading.

Evaluation of Irradiated Fuel Capsules. – The capsules removed from the eight group-II ORR experimental assemblies were examined visually and photographed. These capsules consisted of hollow UO_2 pellets clad with stainless steel. A dark spot, not unlike oxide in appearance, was observed on the wall of capsule 02-2, about 1 in. below the top cap. Annular rings at the pellet junctions were readily visible on capsules 02-2 and 08-2 and faintly visible on capsule 06-2. These three capsules, along with capsule 05-2, were irradiated at a wall temperature of 1600°F, while the other four capsules were irradiated at 1300°F. The foil thermocouple retainer harness was self-welded to the wall of capsule 05-2 in several places. All other thermocouple retainers, which serve as flux monitors, were recovered intact. Diameter measurements of five of the eight NaK containers indicated

general strain, with a maximum increase in average diameter of 0.008 in. at the center of capsule 08-2.

Examinations of photomicrographs of the fuel from capsules L-16xa and L-18xa, which were irradiated in the LITR, have shown an increase in grain size across the pellets from the outer surface to the hotter inner surface. Some columnar growth and grain reorientation are also evident.

Closed-Cycle Experiment ORR-B9. – Irradiation of an experimental assembly containing hollow pellets of UO_2 in coated graphite capsules was terminated at the end of the recent reactor cycle, and the assembly was removed. The capsules had received an integrated dose of 2.2×10^{20} neutrons/cm^2 at a coating temperature of 1500°F. There was evidence of fission-gas release during the final thermal cycle before removal of the capsules from the reactor.

GC-ORR Loop No. 1. – Gas-cooled loop No. 1 was operated on full automatic control in the ORR for extended periods at a pressure of 45 psig with a water-cooled cold finger in the in-pile section. All instrument and control checkouts possible under these conditions have been made.

GC-ORR Loop No. 2. – Construction work on the stainless steel liner adjacent to the south facility plug was carried out on a 24-hr basis during the ORR shutdown of September 6 to 13. The construction is on schedule, and plans have been made for completing this installation during the extended reactor shutdown which began on September 25, 1960.

Fuel Element Fabrication Development. – The use of one-eighth- and one-quarter-hard stainless steel tubing has been proposed as a means for minimizing the grain growth associated with the high-temperature brazing of the midplane spacers to the EGCR fuel tubes. Samples subjected to typical brazing cycles were prepared and evaluated.

Metallographic specimens of spacers brazed onto one-fourth- and one-eighth-hard capsule tubing were examined and compared with similar brazes on annealed tubing. No significant difference in grain size was observed between any of the specimens. This indicates that, for the particular brazing cycle of interest, the degree of cold work in the tube wall has only a minor effect.

Mechanical Property Tests on EGCR Graphite Sleeves. – Fracture surfaces of samples from graphite sleeves containing manufacturers' defects were examined visually in order to estimate the approximate size of the defects. Ring specimens showed an apparent relationship between the size of defects and rupture strength. It appears from the data that any defect (inclusion) that is larger than 1/2 in. long, 1/4 in. wide, and 1/8 in. deep, or a crack as large as 1 in. long by 0.1 in. deep, will reduce the strength to about 1650 psi from a normal value of about 2000 psi.

EGCR Pressure Vessel Design Evaluation. – In evaluating the experimental data relative to stresses on the nozzles of the EGCR pressure vessel head, it was found that additional tests were required. Therefore the model was again subjected to an internal pressure loading, and the strain gages on the nozzles were read. Following this, new gages were mounted along four axial lines on the central control rod nozzle, and another small experimental loop nozzle was instrumented. These additional gages were then read for the same internal pressure loading. All the experimental

data have been reduced and recorded on the drawings which show the strain-gage locations.

The data giving circumferential and tangential stress variations on the inside and outside surfaces of the head were plotted. Separate plots were made for the internal pressure loading and the axial loads on the nozzles. Through the use of dimensional analysis, extrapolation, and superposition, the peak stresses in the head of the reactor vessel were estimated for the following conditions:

1. internal pressure of 350 psi,
2. a service machine load of 15,000 lb on each type of nozzle,
3. axial loads applied simultaneously to the four small experimental loop nozzles,
4. a service machine load and a 350-psi internal pressure loading existing simultaneously,
5. axial loads applied simultaneously to the four small experimental loop nozzles and an internal pressure of 350 psi,
6. an axial load on one large experimental loop nozzle and an internal pressure of 350 psi.

The maximum stress in the head occurs under condition 5 and attains a value of approximately 21,500 psi, excluding thermal stress.

Back-Diffusion Through Labyrinth Seals. – Static and dynamic tests of labyrinth seals have been completed at room temperature (80°F) over the range of pressures from 25 to 250 psig and pressure differentials from 0.5 to 10.0 psi. The data are being plotted and evaluated to determine the seal characteristics at room temperature before continuing with elevated-temperature dynamic tests. Elevated-temperature static tests were completed before the room-temperature dynamic tests were started. No evidence has been obtained thus far of back-diffusion of water into the air, which, at present, is being used as the coolant gas. A theoretical analysis of back-diffusion with helium has indicated, however, that the diffusion factor for helium is 15 times greater than that for air, which could result in an undesirable degree of back-diffusion with helium when operating under certain conditions. It is planned to determine these conditions by conducting selected tests with helium as the coolant gas. The results of a theoretical analysis of flow through labyrinth-type seals, which was programmed on the Oracle, correlate to a fair degree (within about 10%) with the flow values measured in these back-diffusion tests.

THERMAL-BREEDER REACTOR PROGRAM

Homogeneous Reactor Program

Homogeneous Reactor Test. – The core system of the HRT was back-flushed to remove debris which resulted from the core repair work. Partial plugging of the filter basket which was used to collect the debris reduced the core circulation rate by approximately 50%. The fact that most of the debris had been removed was established by doing a second back-flush (with

a new filter basket) in which the circulation rate remained essentially normal. Relative radiation levels of the two filters were consistent with this indication.

The back-flush equipment was removed, and a new circulating pump and multiple hydroclone were installed in the core system. The new pump has crossover pipes, so that flow through the core vessel will be from top to bottom.

Reactor maintenance and alteration activities are being completed; testing will begin early in October.

HRT Chemical Pilot Plant. – The HRT solids-separation system was operated during the core back-flush to collect any fines not removed by the filter screen. Draining and rinsing of the underflow receiver for subsequent dissolution and analyses of the dissolver solution were delayed because the underflow-receiver drain line plugged. The line was unplugged with 75 psi back-pressure after the hydroclone feed line was blocked with a temporary dry-ice freeze plug.

Methods of providing secondary containment around the off-gas charcoal beds were investigated. Operation of the beds below atmospheric pressure, which appears feasible, would greatly minimize the hazard involved and, in conjunction with a vent line (to the stack) from the concrete pit which houses the beds, should provide adequate safety.

Thorium Blanket Studies. – Ten fragments of uranium-thorium oxide gel (5% U based on Th) calcined at 1250°C in air were selected for approximate sphericity and for nominal diameters between 1/8 and 1/4 in. When these fragments were subjected to attrition for 6 hr in the spouting-bed test, the attrition loss varied from 2.1 wt %/hr initially to 0.3 wt %/hr over the sixth hour. The total 6-hr attrition loss was 6.1%.

Microscopic examination of the fragments after the test indicated that most of the weight loss was due to grinding-off of sharp edges and corners. It is expected that more nearly spherical particles of the 1250°C-calcined gel would have attrition rates which are at least as low as the rates obtained with 1750°C-calcined pressed-powder pellets.

In flame denitration of 1 M thorium nitrate solution containing $Al(NO_3)_3$, rounded ThO_2 particles of 1.0 μ average diameter were produced containing only 0.5 wt % Al_2O_3 when the reflector temperature was 1600°C.

Metallurgy and Ceramics. – Approximately 4 kg of thoria pellets were prepared by using a modified D-40 oxide and a single-action Stokes model E press. The D-40 oxide was modified by calcining to 1425°C, then wet ball milling, leaching with hydrochloric acid, drying to 400°C, granulating with 2% Carbowax, and sizing to −35 +100 mesh. Pellets were formed at green densities ranging from 6.2 to 6.7 g/cm^3, fired to 1450°C, and then wet-tumbled to remove the roughness left by the die and punches at the juncture between the hemispherical ends and the cylindrical body of the pellet. The pellets were dried and fired at 1650°C to produce a final density of 9.0 to 9.3 g/cm^3.

Pellets pressed to a green density greater than about 6.3 g/cm^3 showed lamination cracks between the upper hemisphere and the cylinder body, while pellets pressed to a significantly lower density lacked green strength. Pellets fired to only 1300°C before wet tumbling exhibited inferior abrasion resistance, but preliminary evidence indicates that firing to 1425°C

rather than to 1450°C will produce pellets which will behave better during the tumbling operation.

Out-of-Pile Solution Corrosion Tests. – The first runs in the high-temperature, high-pressure titanium loop were completed and demonstrated the adequacy of the loop and the control system. Dilute uranyl sulfate solutions were circulated at temperatures as high as 360°C and with pressurizer temperatures as high as 365°C; under the latter conditions the total pressure on the system was 2925 psia. Before other runs are attempted, the loop will be equipped with a feed and letdown system so that large enough quantities of the second liquid (heavy) phase can be collected in the underflow pot of the hydroclone for analysis. At present the volume of the loop is so small that not enough of the heavy phase is formed to be isolated.

Samples of the light phase were removed from the loop at high temperature, and the following table indicates the changes that were observed in the solution at high temperature:

	Concentration (g/liter)				H_2SO_4 Concentration (N)
	U	Cu	SO_4	Ni	
Original	10.6	1.39	11.10	0.74	0.076
354°C	3.14	1.00	7.40	0.39	0.083
360°C	2.52	0.89	6.85	0.30	0.084

Supporting Radiation Corrosion Studies. – Batch autoclave tests of sorption of uranium and sulfate by bulk samples of hydrous zirconia were continued. One series of tests at 280°C, in which hydrous zirconia prepared by air-drying for 2 hr at 110°C was exposed to a UO_2SO_4 solution containing 40 g of U per liter with 0.02 m H_2SO_4, showed a dependence of the degree of sorption on the length of autoclaving time. About 330 mg of U per g of ZrO_2 was sorbed in 10 min. The uranium sorption gradually decreased with time to about 220 mg sorbed per g of ZrO_2. The sulfate sorption from solution decreased from about 100 mg per g of ZrO_2 in 10 min to 60 mg/g in 2 hr or more. These values do not include the sulfate impurity in the oxide: 39 mg of SO_4 per g of ZrO_2.

A similar series of tests with UO_2SO_4 solution containing 30 g of U per liter was made with oxide prepared by autoclaving with water for 4 hr at 300°C and air-drying for 2 hr at 110°C. About 54 mg of U per g of ZrO_2 was sorbed on autoclaving for 1 hr or more. A 1/2-hr run indicated a sorption of 69 mg of U per gram. The sulfate sorption was about 6 mg/g in all cases.

The decrease in sorption on the 110°C-air-fired oxide with time of autoclaving is believed to result from the decrease in surface area which has been shown to occur during autoclaving. The smaller or possibly negligible change in degree of sorption with time in the case of the 300°C-water-fired oxide is consistent with the small or negligible change in surface area expected on autoclaving this oxide.

The values for the sorption of uranium on the air-fired oxide are based on weight of ZrO_2 and are much greater than those reported by Banter for 250°C sorption. However, Banter's values were based on the weight of charged oxide and are not directly comparable with the present results. It has been

found in the present work that the air-fired oxide sorbs water upon standing in air and was about 50 wt % water when used.

It has been found that uranium sorption at 300°C on the air-fired oxide is somewhat less than that at 280°C for solution concentrations below about 15 g/liter. Above this concentration the sorption is about the same or somewhat greater than that at 280°C.

In-Pile Blanket Materials Tests. — The 5-gpm in-pile slurry loop installed in LITR beam hole HB-2 has operated under irradiation at full (3-Mw) reactor power for 1310 hr, with a total operating time at 280°C of 2511 hr. Operation is being continued. An oxygen atmosphere is maintained in the system. The loop was initially charged with thoria-urania slurry containing 0.5 wt % enriched uranium based on thorium, at a concentration of 1350 g of Th per kg of D_2O (980 g of Th per liter at 280°C).

Loop operation has appeared satisfactory during the irradiation. The flow rate through the filter to the pressurizer has remained steady after declining to a level of about 0.4 cc/sec. Loop control and response remain adequate. The partial pressure of radiolytic gas has remained below 10 psi, indicating a satisfactory recombination rate. Five in-pile slurry samples have been taken and submitted for analysis. The generalized in-pile corrosion rate of stainless steel based both on corrosion-product analysis and on oxygen partial-pressure measurements is less than 1 mil/yr, comparable to rates observed during the out-of-pile preirradiation period.

Evaluation Studies

Thorium-Breeder-Reactor Evaluation Studies. — Further work on the gas-cooled heavy-water-moderated reactor using fuel elements of UO_2 dispersed in graphite plates indicates that a fuel yield of about 4% per year is possible at fuel cycle costs not exceeding 1.5 mills/kwhr. It does not appear possible to reduce the fuel cost to less than 1.2 mills and still maintain a positive breeding gain.

Calculations concerning a polyphenyl-cooled heavy-water-moderated reactor using fuel elements formed by dispersing UO_2 in graphite plates indicate that this system is inferior to the corresponding gas-cooled version, mainly because of the limitation on heat flux imposed by burnout conditions.

The CANDU power-reactor concept was evaluated on the basis of present ground rules and economic conditions. If the fuel fabrication costs (comprising cost of prepared UO_2 powder, cost of pelleting and capsulation, cost of Zircaloy tubing, and cost of fabrication and inspection) do not exceed $50 per kg of UO_2, then the estimated minimum total fuel cycle cost is 2.0 mills/kwhr.

MOLTEN-SALT REACTOR PROJECT

MSRE Design. — Detail drawings of the MSRE core are being prepared. The only change in the core involves the method of construction of the thin metal cylinder which serves the dual purpose of a structural support member and a flow baffle. The cylinder is now mechanically fastened, by bolting,

instead of being welded. The welding appeared to introduce intolerable shrinkage problems.

Stresses in the heat exchanger were analyzed and found to be acceptable. Preliminary specifications for the unit were prepared, and invitations to bid will be sent to manufacturers by October 1, 1960.

Final layout drawings of the primary system were issued. The primary cell is being designed to withstand a positive pressure of 28 psig and will provide absolute containment.

The drain system for the fuel is now 90% detailed. A detailed analysis of the heat removal system for the drain tank was issued. In connection with the fuel drain, a change was made to eliminate a long stagnant-salt line between the reactor vessel and the drain tank. From the reactor inlet volute a 3/4-in. line is taken off and connected to a concentric line inside the 2-in. drain line of the reactor vessel. This inside line discharges fuel at the drain-tank end of the drain line. This fuel then flows back to the reactor bottom plenum through the pipe annulus. A constant flow of fuel therefore circulates in the drain line at all times.

Design of the pumps is proceeding. Specifications for the motor were written, and shielding was designed to protect the pump bearings. The basic design of a freeze flange was completed, and the stresses in the flange were analyzed. The design meets code requirements as far as matching the piping of the system is concerned.

A layout of the secondary containment area, showing air locks for personnel and equipment, decontamination areas, and maintenance control room, was completed. Air handling equipment for the area was specified. Crane and manipulator specifications were written for equipment within the area. Detailing of the secondary area structure is in progress.

The schematic layout of the off-gas system was finished, and some sizing of piping and components was completed.

Detail drawings of a sampler-enricher mechanism are 75% complete. This item will be thoroughly tested in an experimental rig before the final design is made.

The shrinkage to be expected of the graphite in the neutron flux has been reported (Effects of Graphite Shrinkage in MSRE Core, ORNL CF-60-9-10). The tendency of the graphite to bow due to unequal shrinkage will be restrained by molybdenum bands surrounding the core.

Component Development. — Construction of the freeze-flange thermal-cycling facility is approximately 95% complete. A resilient clamp ring for use with one of the test flanges in the thermal-cycle facility was designed and fabrication was started.

Dimensions were computed for 6-in. freeze flanges capable of withstanding externally applied axial loads of 170,000, 110,000, and 70,000 lb. These loads correspond, respectively, to code-calculated allowable piping loads for 6-in. sched-40 pipe and for 6-in.-OD, 0.203-in.-wall tubing and to an estimate of the maximum load that might be induced thermally in the piping. The 110,000-lb flange was selected for further detailing.

A test program was begun to evaluate manipulator-operable disconnects for auxiliary reactor service piping, including off-gas, purge gas, cooling air, lubrication, and other miscellaneous services. Two designs were prepared and fabrication for test was started.

A metal-alloy freeze valve for use with the sampler-enricher access line is being tested for leak-tightness and length of useful service. The valve operates at 1500°F, which is 60°F above the melting point of the 72% Ag–28% Cu alloy used to provide a seal. The leak rate through the valve with 30 psi differential pressure was less than 10^{-8} cc of helium per second for four successive operations. Testing is continuing.

The salt freeze-valve test facility is 95% complete.

The one-fifth-scale MSRE core model was operated with water at 50 gpm. Visual observations indicated that the design of the volute, annular orifice, and core-wall cooling annulus is adequate.

The water velocity is approximately 15 fps at the vessel inlet and appears to taper off to some lower value around the volute. The lowest value is as yet unknown but appears sufficient to cool the volute. The circumferential velocity component in the core-wall cooling annulus was found to be approximately 5.6 fps at the top and 4.6 fps at the bottom, as determined by measuring the slope of the streamlines in the annulus and the flow rate.

The radial pressure drop from the center line to the outer edge of the lower vessel head was found to be 11 in. H_2O without swirl killers. The first design of swirl killers reduced the pressure drop to 3.5 in. H_2O.

A frozen-lead seal around the shaft of a small centrifugal pump has operated continuously for 20,000 hr. The pump is mounted vertically over a fractional-horsepower motor and circulates molten salt at 1200°F.

A graphite-molybdenum compatibility test section was designed and is being fabricated for incorporation into a thermal-convection loop. The test piece includes molybdenum in direct contact with graphite and INOR-8 in the presence of molten salt to test for carbonization and self-welding.

Pump Development. — Mechanical and hydraulic designs were prepared for the MSRE primary and secondary pumps. A single-rotary-element design capable of operating and mounting either the 8 × 6 (primary) or the 6 × 5 (secondary) impellers will be used. The pump tank is designed to accommodate either the primary or the secondary volute and to contain the thermal expansion of system fluid in either circuit. The conventional bearing housing having an impeller overhang of 26 in. was selected; however, its use gives rise to a probable limitation in the range of the resistance to flow in the primary circuit. A preliminary estimate of this range is 45 to 55 ft of head required at 1450-gpm flow. Preliminary computations indicated that 34 in. of nuclear shielding (measured in the axial direction) will be required for the primary pump. The region below the pump inlet will contain 24 in. of the shielding, and the remainder will be contained in the region above the liquid level in the pump tank and below the lower bearing and shaft seal. An order was placed for impellers and volutes cast in INOR-8. Specifications for the drive motor of the primary pump are receiving final review. Design of the primary pump for the reactor is nearing completion. Design of the hot test loop was started.

The molten-salt pump containing a salt-lubricated journal bearing continued in operation with $LiF-BeF_2-UF_4$ (62-37-1 mole %) at 1225°F and has accumulated 3750 hr running time, including 65 starts and stops.

The PKP pump was placed in operation with $LiF-BeF_2-ThF_4-UF_4$ (65-30-4-1 mole %) at 1225°F. The back-diffusion characteristics of a pump-shaft sweep-gas arrangement similar to that planned for the MSRE pumps will be tested with helium as the down-the-shaft sweep gas and with Kr^{85} diluted with helium in the pump-tank gas space.

Chemistry. – The MSRE fuel composition $LiF-BeF_2-ZrF_4-ThF_4-UF_4$ (70-23-5-1-1 mole %), which includes ZrF_4 as an oxide scavenger, was adopted without any experimental investigation of the quinary system. Expectations based on related ternary and quaternary systems were sustained, however, by the recently completed preparation of the first 250-lb batch of the experimental fuel without evidence of unusual behavior. The melting point appears to be less than 445°C, and the first phase to precipitate on cooling has been tentatively identified as an incongruently melting $LiF-BeF_2-ZrF_4$ compound of unknown formula. The vapor pressure of the fuel is low, probably not exceeding four times that of LiF at the same temperature.

Recent experiments carried out in the LiF-NaF eutectic melt show that precipitated UO_2 can be effectively dissolved by adding CrF_3, CrF_2, or NiF_2; the reaction involved seems definitely not to be a simple metathetical exchange of oxide.

The intercalation of graphite by $FeCl_3$ can be prevented by dilution with NaCl. The proportions of NaCl which are barely sufficient for this purpose are 42 mole % at 270°C and 36 mole % at 300°C. Treatment with strong reducing agents, such as Zr^0, appears to diminish the contact angle (less nonwetting) between fuel solvents and graphite.

Metallurgy. – Experiments to develop techniques for producing reliable solidified metal seals for remote operation were continued. The unsintered molybdenum fiber compact which had been impregnated with the gold-copper alloy and used to make a seal between molybdenum plates was successfully opened and closed 20 times. After the eleventh cycle there was some lack of wetting, but this problem was solved by sealing once in hydrogen instead of in helium or argon. This seal is now undergoing metallographic examination.

A sump-type seal, which was fabricated from INOR-8 and had completed ten helium leak-tight sealings, failed on the eleventh try. The failure occurred in the thick-walled sump and allowed alloy to leak out. It is believed that this failure occurred primarily from the large thermal stresses and overheating associated with the induction heating process. This seal is also undergoing metallographic examination.

Tensile tests were completed on cast INOR-8 material for the purpose of establishing design strength values. Low-strain-rate tensile tests on wrought material are in progress to determine the rate of work hardening up to 2% total strain. These data will be employed in the stress analysis of the freeze flange in the MSRE. Data obtained at an extension rate of 0.002 in./min agree with previously published results from tests at the conventional extension rate of 0.05 in./min.

Specimens from the wall of a large graphite pipe, 3-11/16 in. OD, 2-5/8 in. ID, and 6 in. long, were submerged for 100 hr in $LiF-BeF_2-ThF_4-UF_4$ at 1300°F under 150 psig, the standard permeation screening test. The pipe was identified as CEY graphite; however, 5.5% of its bulk volume was permeated by the salt, which is approximately five times the maximum found for the standard size of 1-1/4 in. OD × 7/8 in. ID for CEY graphite. Additional samples of the large pipe are being sought for additional tests in order to determine if the increased porosity resulted from the CEY graphite being fabricated in pipe as large as this.

Further, standard permeation tests have been made on 1-1/4-in.-OD, 7/8-in.-ID CEY graphite pipe. The specimens were taken from the same lot

of graphite as were other specimens for previous tests, but from different pipe. The bulk volume of the graphite permeated by the above salt was 0.3%, compared with the 1.0% that was obtained on the specimens from another pipe in the earlier tests. Additional tests are planned to determine the permeability range for this lot of CEY graphite.

Corrosion Tests. — Corrosion pump loops, eight of INOR-8 and two of Inconel, have been in operation with various mixtures of fluoride salts during the past two months. These loops operate at temperatures from 1200 to 1500°F with a ΔT of 200°F. They have accumulated from 3700 to 20,000 hr at operating conditions. Operation of the two Inconel loops was terminated in this period for metallurgical examination after 13,000 and 15,000 hr of operation.

In-Pile Tests. — Two graphite-fuel capsule experiments were irradiated in the MTR to determine the effect of radiation on the penetration of molten-salt fuel into graphite and the degree of fission-gas retention by the graphite. The capsules were filled with $LiF-BeF_2-UF_4-ThF_4$ (67-18.5-0.5-14 mole %) and were mounted in a sodium-filled tank. The estimated fuel temperature was about 1300°F. The power density of the fuel was 200 w/cc, and the volume of fuel per capsule was approximately 18 cc. The first experiment (ORNL-MTR-47-1) ran for 700 hr and the second experiment (ORNL-MTR-47-2) for 1500 hr.

The two experiments were disassembled at Battelle Memorial Institute during August 1960. All capsules in the first experiment and three of the four capsules in the second had failed. It is believed that the failures were initially stress failures caused by the freezing and melting of the fuel within the capsule during the power cycles of the reactor and that the subsequent attack on the bellows was due to an interaction between fuel, sodium, and the capsule material.

The good capsule contained type S4A graphite. Analysis and examination of that sample is proceeding at BMI. External appearance of the graphite is good, and the physical dimensions indicate changes in the order expected. The weight gain of the graphite corresponds favorably to results from out-of-pile tests for the same type of graphite. From the weight increase the volume per cent of fuel penetration was 0.71 at 100 psia for the S4A test specimen, whereas an out-of-pile test at 150 psia indicated a volume per cent penetration of 1.0.

Reactor Analysis. — The induced activity in INOR-8, the primary fuel salt, and the secondary coolant salt of the MSRE was calculated. Both (n,γ) and charged-particle reactions were considered. The total activity associated with INOR-8 following 1 yr of irradiation was 2.2×10^{12} Mev sec^{-1} cc^{-1} for material exposed to the average core flux. Charged-particle reactions contributed more than 50% of the activity.

The activity of the fuel leaving the core was 3.8×10^{10} Mev sec^{-1} cc^{-1}, and the activity of the secondary coolant entering the radiator pit was 4.8×10^{8} Mev sec^{-1} cc^{-1}. Both activities were due primarily to the high-energy gammas following the $F^{19}(n,\alpha)N^{16}$ reaction; the neutron flux used in these calculations was that associated with no shielding between the reactor and the region of interest.

HIGH FLUX ISOTOPE REACTOR

Critical Experiments. – Critical experiment 1 was terminated. Critical experiment 2 will utilize an actual HFIR fuel element. No further report will be made until the fuel element and the assembly have been constructed.

Corrosion. – A corrosion test, simulating the hot channel condition in the HFIR and utilizing a new type of specimen that permitted examination of water-cooled surfaces before and after test, was successfully completed. The conditions were: heat flux, 1.4×10^6 Btu ft^{-2} hr^{-1}; coolant temperature, 188°F inlet and 219°F outlet; flow rate, 40 fps; water pH, 5 (HNO_3); alloy 6061-T0; duration, ten days. During the test the maximum rate of metal temperature increase near the outlet of the specimen was 5.4°F per day. Examination of the exposed surfaces of the specimen at the conclusion of the test revealed no evidence of localized attack; a thin, transparent oxide film with a maximum thickness of 0.0005 in. covered the surfaces of the specimen. Although a complete determination of the extent of corrosion damage has not been made, it appears that the maximum penetration was less than 0.001 in.

From this test and other similar ones, it appears that the corrosion rate of the aluminum cladding on the fuel elements will be sufficiently low to prevent appreciable penetration of the cladding and to prevent the formation of heavy oxide films which would result in excessive temperature in the fuel plates. Future tests will include an examination of the effect of pH and of the effect of lowering the system overpressure to allow film boiling and alternate wetting of the corrosion film. Experiments with a duration of more than ten days are planned to investigate longer fuel cycles and conditions to be experienced in the target area.

Metallurgy: Fuel Element Core Preparation. – An investigation has been initiated to determine the variables which affect boron losses during the preparation of aluminum-base core alloys.

A series of powder-metal fuel cores were pressed from three different particle ranges of aluminum powder: –100 mesh (as received and blended), –100 +325 mesh, and –325 mesh. All the cores made in this series contained 38.54 wt % U_3O_8, 64.10 wt % aluminum, and 0.065 wt % B_4C. Precautions were taken to eliminate variations due to pressing.

The results show that greater consistency is obtained with a smaller size range of aluminum particles. Both the –100 +325 and the –325 mesh showed good reproducibility, while the as-received –100 mesh aluminum showed erratic results when used in tapered cores. Further, the results show slightly less taper than the required 50 to 200 mils. A new die top with a greater angle is being produced to compensate for this.

Metallurgy: Fuel Plate Fabrication. – Approximately 95 HFIR fuel cores produced by forging techniques and containing 30 wt % U, 0.03 wt % B, and 2 wt % Si were measured for density variations by a water displacement technique prior to assembly into billets. The average density was 3.62 ± 0.040 g/cc, based on 84% of the measured values. The other 16% were beyond these limits. Correlations between density measurements and uranium analyses indicated that the addition of silicon to this alloy increased the apparent density of the uranium-aluminum compound from 6.80 to approximately 7.12

g/cc. These marked variations from the calculated density of UAl_3 may be attributed to silicon segregation in the alloy and the formation of a complex uranium-aluminum-silicon intermetallic compound.

Numerous blisters have occurred in HFIR fuel plates during fabrication and subsequent heat treatment at 500°C. Of 92 plates fabricated to date, 65% blistered. The blistered areas are being examined metallographically to determine the location and possible cause of the blisters.

Metallurgy: Fuel Plate Forming. — Performance of the refinished Marforming die for making external fuel section plates was checked by the production of five test plates of solid 6061-0 aluminum and ten dummy fuel plates containing a 30 wt % U-Al core. The dimensions of these plates indicate the die to be satisfactory for the production of solid 6061-0 fuel plate. However, poor duplication and a pronounced saddle were experienced in the plates containing cores. Similar-symmetry measurements taken from the plates containing cores show a spread of 14 mils, whereas duplicate points measured on the solid aluminum plates show a spread of 5 mils.

Metallurgy: Corrosion Specimen Fabrication. — The fabrication of heat-flux corrosion specimens has necessitated the welding of a 0.025-in. section of 6061 aluminum. By adding 1100 aluminum as a weld diluent and using electron-beam welding, smooth, high-integrity welds with minimal shrinkage have been produced which are adequate for the required service in hot, high-pressure water. Heliarc welding techniques have also been developed to produce these specimens.

Design: Fuel Element. — The fuel element was modified to include a 3/8-in.-thick water gap between the inner and the outer fuel rings to provide for increased neutron moderation in this area. At the same time a decision was made to increase the fuel plate thickness from 40 to 50 mils. While there is no clear evidence that the 40-mil thickness is unsatisfactory, the 50-mil thickness is more conservative by virtue of the factor of 2 increase in plate stiffness. The water-gap thickness of 50 mils between plates is unchanged.

The increase in plate thickness reduces the heat transfer area per unit volume and the free area for water flow. To regain the heat transfer area the fuel-bearing portion of the element was lengthened from 18 to 20 in. To maintain similar exit water temperatures with the new dimensions, the design flow velocity in each channel was increased from 40 to 42 fps. The new fuel plate surface temperature of 346°F at the hot spot is about the same as for the 40-mil plate.

Design: Beryllium Reflector. — The beryllium reflector was redesigned to accommodate the new fuel element outside diameter of 17-1/8 in. To reduce the cooling water requirements an allowable thermal stress of 12,000 psi (MTR design value) was used rather than the more conservative 7000-psi level previously considered. The design concept remains unchanged, that is, an inner removable beryllium reflector section consisting of several concentric cylinders separated by 50-mil coolant annuli, and an outer permanent beryllium annulus composed of upper and lower 1-ft-high sections with cooling provided by vertical coolant holes.

NUCLEAR TECHNOLOGY AND GENERAL SUPPORT

Fuel Element Catastrophe Studies. – The results of many previous experiments on the release of fission gases from UO_2 have shown an initially high release, followed by a slower release at a uniform rate. Some diffusion experiments of this type were conducted in helium with PWR-type sintered UO_2. Typical release values obtained by heating UO_2 at 1100°C for 5 hr in flowing helium (1.63 ppm oxygen) without prior evacuation of the furnace were: rare gases and iodine, 1%; tellurium, 0.8%; and cesium, 0.06%. At the same time 3% of the UO_2 was oxidized to U_3O_8. In a separate set of experiments (at the same temperature and for the same time) with prior evacuation of the equipment and with helium that had been passed over molten lithium, there was no oxidation of UO_2 and the fission product release values were: rare gases, iodine, and tellurium, 0.2%; cesium, 0.02%; strontium, 0.003% or less. Diffusion constants and activation energies were calculated from the rate of the slow, uniform release, and in the temperature range 1000 to 1500°C, D values of 10^{-17} to 5×10^{-14} cm^2/sec and an activation energy of 91 kcal/mole were obtained. From the gross release values (initial rate included), slightly different D values of 10^{-16} to 10^{-13} cm^2/sec and an activation energy of 96 kcal were calculated. Diffusion coefficients calculated from the reduced rate of release in purified helium corresponded to the lower range of values.

The usually anticipated result of a loss-of-coolant accident in a UO_2-fueled reactor is core meltdown; however, some circumstances might lead to loss of cladding with or without resulting oxidation of UO_2 by air. In a series of experiments in each of which a UO_2 pellet was heated 1.5 hr in air at a temperature in the range 400 to 1500°C, the extent of oxidation of the UO_2 and the release of individual fission products were measured. The results show maxima in degree of oxidation and release of iodine and rare gases at about 600 and 1400°C, with a minimum at 900°C. The other fission products measured, ruthenium, tellurium, and cesium, do not show comparable variations since their release rate increases regularly with temperature. The explanation for this behavior is believed to lie partly in the decrease, with increasing temperature, of the free energy value for the reaction of UO_2 and O_2. Typical release values at 900°C in the region of the minimum are: tellurium, 1%; cesium, 0.5%; ruthenium, 5%; rare gases, 7%; iodine, 8%. At 1400°C the corresponding values are: tellurium, 30%; cesium, 11%; ruthenium, 98%; rare gases, 13%; iodine, 22%. Strontium release values are essentially nil ($\sim$ 0.005%).

Reactor Evaluation Studies. – The Expire-Epitaph program, which calculates reactivity lifetime and fuel cost, has been completed and checked out. Equilibrium plutonium recycle calculations have been initiated on three reactor types. For the gas-cooled reactor the optimum uranium enrichment (for minimum fuel costs) for the first cycle varied from 2.2 to 2.6%, depending on the value of plutonium. For a uranium enrichment of 2.4% the reactivity lifetime was 11,900 Mwd/ton and the fuel cost was 1.1 mills/kwhr (thermal) for a plutonium value of $12/g. The plutonium resulting from the first cycle was blended with uranium of various enrichments. The optimum uranium enrichment for the second cycle varied from 2.5 to 2.7%. For a uranium enrichment of 2.6% the reactivity lifetime was 15,000 Mwd/ton and the fuel cost was 1.37 mills/kwhr (thermal) and nearly independent of plutonium value.

Zirconium Metallurgy. — Recent studies on the kinetics of the transformations of the Zr-Nb-X type of zirconium-base alloys of interest to the HRP for construction of the fuel containment vessel have shown that increased quenching temperatures, increased homogenization times, faster quenching rates, and shorter aging times at room temperature after quenching decrease the incubation time for the formation of omega phase from the retained beta phase on aging at 400°C, and increase the amount of omega phase present at any given time interval after the start of omega-phase formation. All the variables studied should affect the concentration of vacancies present in the alloy at aging temperature in a manner which would indicate that an increased concentration of vacancies increases the rate of nucleation of omega phase. Resistometric studies of the transformations of the Zr—15% Nb alloy tend to confirm the previously determined kinetic data in the following respects: maximum rate of formation of omega phase occurs at 400°C, omega phase formed at and below 400°C is partially redissolved or reverted on heating to temperatures above 400°C, the reversion reaction of omega phase and the niobium-enriched beta phase to form the original beta phase composition and structure is complete at 540°C, and the M_s temperature for the formation on cooling of the martensitic Widmanstätten phase in a retained beta matrix is between 200 and 250°C. The data indicate that the specific heat treatments required for obtaining and stabilizing the microstructure in the retained beta zirconium alloys to yield the desired mechanical properties in large structural members will depend to an appreciable extent on the massiveness of the part.

Nondestructive Test Development. — A calibration of the gamma scintillation spectrometry equipment has been made for the assay of U^{235} in 18.65% U—Al alloy core blanks, using data from chemical analysis of a number of single core blanks. A heat of 72 blanks was measured, and from this calibration curve it was determined that an average of 10.2 g of U^{235} was present per core. Gamma scintillation measurements in the past have agreed quite well with the chemical analyses, which lends confidence to these values. However, this figure is not in agreement with the average value of 10.53 g/core calculated from the dip analysis made at the rolling mill. To further evaluate the apparent discrepancy, a new heat of 72 cores of 18.65% uranium has been requested. Measurements of volume and density of these cores will be made to permit correction for the amount of aluminum. Successful completion of the analysis of this new batch should complete the first phase of this program and allow routine assays of U-Al core blanks having concentrations within the range of the calibrations.

Fuel Cycle Technology. — Fragments of uranium-thorium oxide (5% U based on Th) calcined at 1200°C, with a particle density of 9.8 g/cc and 85% of its particles > 1.7 mm, were prepared by a simplified procedure consisting of addition of $UO_3 \cdot H_2O$ to thoria sol followed by settling, decantation of excess nitrate, drying to gel, and prefiring to 500°C.

Thorium oxide containing 7.1 to 4.8 g of residual volatile matter (nitrate + H_2O) per 100 g of ThO_2 was prepared by thermal denitration with steam or air in a batch rotary calciner at 340 to 390°C for 12 min. The oxide was dispersed by water to sols from which dense thoria gel fragments could be made. Steam-denitrated oxides were superior to those denitrated in air. They were more readily dispersed to sols and produced larger dense

fragments when converted to calcined gels. Steam denitration was easily controlled and gave an off-gas which was completely condensable by cooling.

Reactor Controls Development. — The time response of the ORNL period safety system has been studied analytically and experimentally. It is now clear that, for the case of short periods ($\tau < 1$ sec) at low levels, the scram response is determined entirely by the input capacitance; in particular, for $C = 10^{-9}$ f the period scram cannot be initiated at currents below 8×10^{-11} amp, approximately six decades below full reactor power. At all periods between 5 msec and 1 sec, the scram is initiated at least four decades below the full power of the reactor, thus extending substantially the range of a fast safety system. The effect upon the scram response of a steady current superimposed upon the exponentially rising current has been studied. A steady current of 10^{-11} amp has no effect whatever; 10^{-10} amp has only slight effect at periods between 5 msec and 1 sec. A steady current of 10^{-9} amp slows the scram response only at long periods (0.1–1 sec) and by an amount which permits less than one decade rise in power before the scram is initiated. A steady current of 10^{-8} amp has a somewhat larger effect at long periods. The results of this study indicate that the role of chamber compensation in the period scram system must be reviewed.

Power Reactor Fuel Processing: Mechanical Processing. — Checkout of the SRE dejacketing facility was continued. The lead screws and companion nuts from both the multipurpose saw and the dejacketing machine were coated with a baked-on lubricant, Bemol No. 120 (MoS_2 in a phenolic resin). Tests showed that this resin is unaffected by submergence in mineral oil and that the lubricant withstands up to 10^9 r of gamma radiation at a temperature of 225°F. The maximum gamma radiation expected from the SRE core 1 fuel is $\sim 10^7$ r. The redesigned auxiliary decladder roll cutters (Carpenter K.W. steel, hardened to 60–62 Rockwell C, 15-mil-deep cutting edge at a 60° included angle) successfully slit 12 SRE jackets from cambered steel slugs without failure or noticeable wear on the cutting edges.

Dissolution rate data on ThO_2-UO_2 pellets (Universal Match Co.) in fluoride-catalyzed nitric acid were used to estimate the effective surface area of partially dissolved pellets. The surface area was found to vary from 1.2 to 2.4 times the initial superficial surface area at up to 80% dissolution of the pellets.

Power Reactor Fuel Processing: Zirflex Process. — In Zirflex decladding studies with 4.5 M NH_4F–0.5 M NH_4NO_3, the average dissolution rate of Zircaloy-2 was decreased only $\sim$10% when a volume of 1.0 M NH_4OH (rather than H_2O) 1.8 times the dissolvent batch volume was gradually added to the dissolvent to maintain a constant level in the dissolver as the overhead condensate was withdrawn. During a batch reflux run the NH_4OH concentration of the condensate was a maximum of $\sim$ 1.15 M after $\sim$ 5 min and then steadily declined; the average NH_4OH concentration for the dissolution period was $\sim$ 0.3 M.

Power Reactor Fuel Processing: U-Mo Fuel Aqueous Processing. — The initial rate of dissolution of uranium–3% molybdenum alloy was measured in boiling nitric acid–phosphoric acid and nitric acid–ferric nitrate solutions. In 8 and 10 M HNO_3 the rate passed through a maximum when the phosphoric acid concentration was 0.25 M; however, at all phosphoric acid concentrations up to 1 M the rate was acceptably high (between 60 and 300

mg min^{-1} cm^{-2}). In 8 and 10 M HNO_3 solutions containing up to 1 M ferric nitrate, the initial rate of dissolution varied between 140 and 310 mg min^{-1} cm^{-2}.

In U-Mo core dissolvents containing ferric nitrate, the corrosion rate of titanium was ≦ 0.1 mil/month over a 620-hr exposure.

Power Reactor Fuel Processing: Zircex Process for U-Mo Fuel. – An oxyhydrochlorination flowsheet for the CPPD-1 fuel (90% U–10% Mo) canned in aluminum was developed and was tested satisfactorily in the laboratory. The major advantage of the process is the removal of over 90% of the molybdenum as volatile molybdenum chlorides before solvent extraction. The operations involve removal of the aluminum can at 300°C with 60% HCl–N_2, core oxidation–molybdenum volatilization at 400°C with 15% HCl–air, molybdenum cleanup at 400°C with pure hydrogen chloride, chloride cleanup at 400°C with 0.6% H_2O–air, and U_3O_8 dissolution in 4 M HNO_3 to yield a stable solution containing 1 M U, 0.017 M Mo, 0.003 M Al, 175 ppm Cl, and 1.7 M HNO_3.

Power Reactor Fuel Processing: Processes for Advanced Fuels. – Disintegration and leaching of a solution-impregnated graphite sphere (pebble bed prototype by Sylcor) with 90% HNO_3 at 25°C resulted in recovery of 99.9% of the uranium, whereas grinding to –10 +20 mesh and leaching with boiling 70% acid resulted in recovery of only 99.3% of the uranium.

Reaction of 1 g of uranium monocarbide (4.00 meq) with water at 90°C in a helium atmosphere yielded 0.55 mmole of hydrogen, 3.58 mmoles of methane, 0.11 mmole of ethane, and 0.03 mmole of propane. The uranium product was an amorphous solid which was at least 98% U(IV), as determined after dissolution in HCl. No nonvolatile carbon was found, but the technique for carbon determination in HCl is not very sensitive. Thus the principal reaction of uranium monocarbide with water at 90°C appears to be $UC + 2H_2O \rightarrow UO_2 + CH_4$.

Beryllium metal dissolved in boiling 4 M H_2SO_4 at a rate of 63 mg min^{-1} cm^{-2}. In boiling 6 M NaOH and 4 M HNO_3–0.05 M NaF, the initial rates of dissolution were 2 and 8 mg min^{-1} cm^{-2} respectively.

Niobium reacted at 600°C with anhydrous hydrogen chloride and hydrogen fluoride at rates of 3.0 and 1.8 mg min^{-1} cm^{-2} respectively.

Power Reactor Fuel Processing: Solvent Extraction Studies. – Tests on the Acid Thorex flowsheet indicated that dilute monobasic aluminum nitrate may be used in the strip solution in partitioning of thorium and uranium. With this salt the carry-over of acid to the uranium strip column and the total amount of aluminum in the second cycle waste are decreased. The distribution coefficients of zirconium between 30% tributyl phosphate in dodecane and 6 M and 4 M HNO_3 were 1.6 and 0.42 respectively. Preliminary experiments showed that there are at least two species of zirconium present in 2 M HNO_3, the more extractable of which has a distribution coefficient of about 0.12.

Power Reactor Fuel Processing: Darex and Sulfex Processes. – In initial dissolution studies on Consolidated Edison ThO_2-UO_2 pellets containing 2 and 4% "fissia" (a simulated fission product mixture), neither zirconium nor molybdenum dissolved in the Thorex reagent, 13 M HNO_3–0.04 M NaF–0.1 M $Al(NO_3)_3$. Uranium and thorium dissolution was also incomplete.

Titanium showed negligible attack during 65 Thorex feed adjustment evaporations.

Fluoride Volatility Processing. — A study of the dissolution of type 347 stainless steel in 37-52-11 mole % $LiF-NaF-ZrF_4$ was made in conjunction with the processing of the EBR core 2 fuel by the fused-salt volatility process. Dissolution rates of 1.9 and 2.1 mg cm^{-2} min^{-1} were found. Favorable corrosion rates, 0.06 mil/hr, were measured for both nickel and INOR-8, indicating possible use of the present pilot plant dissolver for recovery of the EBR fuel.

During dissolution of high-fired ThO_2 in ZrF_4-bearing melts, ZrO_2 precipitated, indicating direct metathesis to ThF_4 and ZrO_2 rather than oxyfluoride formation, as suggested earlier. A volatility flowsheet for dissolution of thoria fuel would thus require use of HF for reconversion of the ZrO_2 to ZrF_4, or prevention of its precipitation in the first place. High-fired UO_2 dissolved rapidly, at a rate > 10 mg cm^{-2} min^{-1}, by hydrofluorination in 31-24-45 mole % $LiF-NaF-ZrF_4$. Metathesis with the ZrF_4 apparently did not occur.

The solubility of NbF_5 in anhydrous tank HF was found to be 5.2 wt % at 10°C and 5.5 wt % at −79°C. The negative temperature coefficient appears favorable in preventing NbF_5 precipitation in the HF recovery system when niobium-containing fuels are processed.

Aluminum sampling cups cannot be used in molten fluoride systems. Analysis of a black residue collected on such cups after immersion in fused salt showed a high concentration of nickel and iron, removed from the salt as a result of reduction of their cations by the aluminum.

A condensation pressure analyzer and a gas density detector were successfully used to monitor the UF_6 concentration in the effluent from an NaF bed that was absorbing UF_6 from an inert carrier gas. As the sorption wave reached the end of the bed, the UF_6 concentration in the effluent rose from < 0.01 to 10 mole %. Gas density detector readings were reproducible to within 1%.

PHYSICAL RESEARCH PROGRAM

Reactor Operations. — The Graphite Reactor was annealed during the weekend of Labor Day. The operation was very successful, and samples taken since the annealing have indicated that the stored energy above the specific heat curve was removed as planned. Regular operation began on September 6 and has been routine since that time.

The ORR is now operating routinely at 30 Mw. For a few weeks after 30-Mw operation was begun there was some trouble with the new equipment, but this has been largely resolved. Two eight-day shutdowns in September were scheduled for installation work on the Gas-Cooled Reactor loop No. 2. It is expected that in October the reactor will begin operating on an eight-week cycle with approximately one week of down time per cycle.

Waste Disposal. — Waste disposal operations were routine. The new seepage trench is taking most of the intermediate-level liquid waste, with a small balance still going to the open pits. Efforts are being made to reduce the volume of intermediate-level waste, and design work has started on a new seepage trench to supplement the first one.

The gas monitoring system on the stacks is in operation and has been useful in detecting a number of small releases of gaseous activity.

Proportional-sampling stations are being installed on White Oak Creek and Melton Branch. These will permit a more accurate determination to be made of the amounts of activity coming from the lake bed and the two streams.

Hot-Cell Operations. — Work is continuing on decontamination of the 3025 hot cells and on preparation for operation. Cell No. 6 is almost ready, but manpower is not yet available for an extensive decontamination program on the other cells.

PHYSICS AND MATHEMATICS RESEARCH

Neutron Diffraction. — Neutron diffraction experiments have been performed on single crystals of dysprosium in an attempt to understand the nature of the magnetic ordering that occurs in this metal at low temperatures. Previous magnetic measurements [Phys. Rev. 109, 1544 (1958)] have shown that dysprosium has two magnetic ordering transitions: at 179°K there is a transition from the paramagnetic state to an antiferromagnetic arrangement of the moments, while at 85°K there is a transition from antiferromagnetism to ferromagnetism.

The neutron diffraction results indicate that the magnetic structure in the antiferromagnetic region corresponds to a helical-type arrangement of the moments. In this arrangement the moments within a hexagonal layer are aligned parallel, with the moment direction perpendicular to the c axis of the crystal. However, between adjacent layers the moment direction within the layer changes by a specific angle, and the value of this angle is dependent on the temperature of the sample. At the ferromagnetic transition there appears to be a sudden change from this helical-type structure to the normal type of ferromagnet, and the moment direction remains perpendicular to the c axis.

Fast-Chopper Time-of-Flight Spectrometer. – In order to reduce the effect of multiple scattering, the capture from the 132-ev cobalt resonance has been remeasured with a cobalt sample only one-third as thick as that formerly used. From these measurements the radiation width of this level was determined as being 0.67 ± 0.15 ev. The radiative capture cross section of tantalum was measured in the range ~ 0.2 to 10 kev, and the data agree to within ~10% with the ORNL pulsed Van de Graaff data. A resonance was observed in zirconium at 183 ev with the capture tank.

Transmission measurements have been made on a sample of Np^{237} (1/N = 520 barns/atom) from about 2.5 to 100 ev with the MII (high resolution) rotor and with $B^{10}F_3$ detectors located at the 180-m flight station. A resolution of 0.011 μsec/m was observed with this equipment in the range 35 to 100 ev. The data are now being analyzed for resonance parameters with the aid of an IBM 704 code.

High-Voltage Experimental Program: The Reaction $C^{14}(p,n)N^{14}$. – The reaction $C^{14}(p,n)N^{14}$ has been studied up to 5 Mev bombarding proton energy with good resolution, using targets of high isotopic enrichment. Levels previously unseen in this reaction were found at proton energies (in Mev) of 3.19 (Γ = 6 kev), 3.38 (Γ = 24 kev), 3.63 (Γ = 13 kev), 3.89 (Γ = 35 kev), 4.19 (Γ = 112 kev), 4.24 (Γ = 27 kev), 4.61 (Γ = 140 kev), and 4.93 (Γ = 106 kev). Excitation curves at three angles in the region of 2.9 Mev proton energy show the effect of the previously known $J = 3/2^-$ (Γ = 80 kev) resonance interfering with a level of opposite parity. Effects of the nearby $J = 7/2^-$ (Γ = 40 kev) level are not seen, presumably because of the low penetrability of the outgoing F-wave neutron. The thresholds for the second and third neutron groups were investigated with a lithium iodide detector. A new threshold, that for production of the third neutron group, was measured as being 4.910 ± 0.008 Mev, in agreement with the known energy of the second excited state in N^{14}.

High-Voltage Experimental Program: Level Structure of N^{14}. – Yields and angular distributions of the gamma rays from the first and second levels in C^{13} following excitation by inelastic proton scattering have been measured. Levels are observed at bombarding proton energies (in Mev) of 3.80 (Γ = 100 kev), 4.1 (Γ = 150 kev), 4.14, and 4.52 (Γ = 150 kev). The first three levels result in excitation of the first excited state, and the last in excitation mainly of the second excited state of C^{13}; N^{14} excitation energies corresponding to these levels are 11.07, 11.35, 11.39, and 11.74 Mev. Possible spin and parity assignments have been made on the basis of the gamma-ray angular distribution of the 4.52-Mev level. The $C^{13}(p,n)N^{13}$ neutron yield has been remeasured, giving better values of the resonant energies. Neutron angular distributions were obtained for several bombarding proton energies.

Spectroscopy Research Laboratory. – The vapor-phase infrared absorption spectra of ZrF_4 and HfF_4 were studied from 3000 to 300 cm^{-1}, using a modified single-beam spectrophotometer. The required temperature range, 700 to 900°C, was reached in the center of a gas-tight infrared cell filled with helium. Two strong absorption bands, at 670 cm^{-1} in ZrF_4 and at 650 cm^{-1} in HfF_4, have been assigned to the asymmetric stretching modes (ν_3) of these tetrahedral molecules.

Solid solutions of polyatomic inorganic ions in alkali halide lattices were obtained by quenching the molten alkali halide in which the appropriate compound was dissolved. Studies of the infrared spectra of these samples have shown that in many cases vibrational absorption intensities of the solute ions are increased appreciably by the formation of solid solutions. This occurs both through narrowing of the absorption bands and through increased dipole moment changes, due to interactions with neighboring ions, during the vibrational motions. The spectra of the OCN^-, CO_3^{--}, and SO_4^{--} ions have been studied in some detail in KCl, KBr, and NaCl lattices, and the applicability of this fusion technique to qualitative and quantitative analyses has been determined. Quantities of the three solute ions in the microgram range can be easily detected and estimated.

CHEMISTRY RESEARCH

Spectrophotometric Studies of Solutions at Elevated Temperatures and Pressures. – The design and development of a high-temperature high-pressure spectrophotometer system by Applied Physics Corporation is proceeding on schedule. The optical design and the design of the control system and console are essentially complete.

The least-squares resolution computer program is being applied to the analysis of the Pu(IV) spectrum in $HClO_4$ from 8000 to 29,000 cm^{-1}.

A new computer program, SPECSYN, was completed. This program synthesizes a spectrum, over any given range, from estimates of the individual band parameters of an experimental spectrum. It then plots the individual bands and/or the resulting synthesized spectrum on the Oracle curve plotter, along with plots of the observed spectrum for comparison on the same picture.

Solvent Extraction Research. – Americium was preferentially extracted from 9 N LiCl–1 N $AlCl_3$ by 0.5 M trilaurylamine in Solvesso 100 by factors of ~100 with respect to the fission product rare earths. This extraction involves an anion exchange with the extractant and is not applicable to curium or the heavier transplutonium elements. However, prior removal of americium would increase the potential usefulness of some processes, such as extraction by tributyl phosphate from concentrated chloride solutions, in which the transplutonium elements are more extractable than the fission product rare earths.

Solvent extraction experiments with radioactive material left after decay of 6.7-day Lu^{177} led to identification of a second isomer of Lu^{177} with a half life of ~200 days. The gamma spectra of the two isomers have at least four peaks in common, but both the beta and the gamma spectra of the new isomer are more complex.

Nitroparaffins, RNO_2, were identified as the nitration products of Amsco 125-82 which contribute to poor decontamination and ineffective solvent cleanup in TBP-Amsco solvent extraction systems. The nitroparaffins, ineffective extractants themselves, undergo tautomeric shift to an acid form, $RCHNO_2 \rightleftharpoons RCNOOH$, which in combination with TBP acts as a strong extractant for Zr-Nb and Ru.

In final laboratory development of the tertiary-amine extraction process for separating technetium, neptunium, and uranium from fluorination

plant residues (ORNL-2874, -2991), a direct pH indicator test proved sufficient for controlled hydrolysis of 98 ± 1% of the amine nitrate extractant, permitting elimination of 98% of the nitrate from the technetium product. Calculations based on this, together with separation factors and distributions from the continuous countercurrent tests reported previously, indicate that with less than 20 stages in the nitrate-removal section this single-cycle process could produce ammonium pertechnetate essentially free from nitrate.

Chemical Engineering Research. – In studies on mass transfer across an interface, careful measurements of interfacial tension between water and tributyl phosphate solutions in Amsco at varying temperatures and uranyl nitrate concentrations showed that interfacial tension increases (1) approximately 0.1 dyne per degree centigrade rise in temperature and (2) nonlinearly with increasing uranyl nitrate concentration.

For a flowsheet in which 5% TBP in Amsco is used to extract uranium from 3 M HNO_3 at a phase ratio of 1/1, the flooding capacity of pulsed solvent extraction columns increased from 300 to 1500 gal ft^{-2} hr^{-1} as the pulse frequency was decreased from 90 to 50 cycles/min. The column contained sieve plates (0.125-in. holes, 23% free area) and was operated with the aqueous phase continuous. Flow capacity was approximately 20% greater for a column that contained nozzle plates (0.125-in. holes, 10% free area) and was operated with the organic phase continuous. Flooding rates for strip columns containing sieve plates and operated with the aqueous phase continuous at an aqueous/organic phase ratio of 1/4 increased from 300 to 800 gal ft^{-2} hr^{-1} as the pulse frequency was decreased from 70 to 30 cycles/min.

Nuclear Methods of Chemical Analysis. – Small concentrations of beryllium (10 μg) have been determined by measuring the prompt-gamma or capture-gamma radiations that result from the interaction of the alpha particles from Am^{241} (452-yr) with the beryllium. The high-energy gamma photons (3.5–4.5 Mev) of almost instantaneous half life that are produced in the nuclear reaction $Be^9(\alpha,n,\gamma)C^{12}$ are specific for beryllium. Although the intensity of their production is a linear function of the beryllium concentration in a sample material, it has been found most practical to use amounts of Am^{241} no larger than 10 mg. All the analyses are completed by gamma spectrometry.

Reactor Neutron Cross Sections. – Effective cadmium cutoff energy calculations were extended to include cylindrical filters with height/diameter ratios as high as 11. Some effective boron cutoffs were also calculated for comparison with values for cadmium cylinders of height/diameter ratios of 1, 2, and 3.

Flux depressions in the vicinity of and within infinite cadmium slabs were calculated with the use of the SNG reactor code on the IBM 704 computer at ORGDP. These results are pertinent to burnout calculations concerning cadmium filters during long irradiations.

An upper limit of 0.1 barn was arrived at for the formation cross section of the 25-sec isomer of Po^{211} from Po^{210} for thermal reactor neutrons.

Chemistry of Technetium. – The visible and near-infrared absorption spectrum of $TcCl_6^{--}$ in 6 M HCl was measured over the spectral range 4000

to 13,850 A. The following absorption maxima were observed, with the molar absorbancy indices given in parentheses: 6150 A (0.50), 7050 A (3.14), 10,490 A (1.24), and 10,960 A (0.99). Two shoulders at approximately 11,200 and 11,600 A were observed which represent unresolved absorption bands close to the 10,960-A band. No vibrational fine structure was observed, a distinction from the $ReCl_6^{--}$ spectrum.

Thermochemistry and Calorimetry. – Heat-capacity measurements on K_2ReBr_6 previously reported as giving a transition of the cooperative type at 15°K indicated the need to extend the measurements on K_2ReCl_6 (previously measured over the temperature range 14 to 320°K) to lower temperatures. Heat-capacity measurements on K_2ReCl_6 from 6 to 20°K, now completed, reveal a cooperative-type transition at 12° which represents the transition from an antiferromagnetic to a paramagnetic state. The revised entropy of K_2ReCl_6 is 88.84 cal deg^{-1} $mole^{-1}$.

Chemical Separation of Isotopes. – Mass spectral studies of the isotopic nitrous oxides were carried out on samples labeled with N^{15}. The cracking pattern was interpreted in terms of an activated complex with a ring structure. Metastable transitions were observed in the mass spectra of all four isotopic species. Studies of pressure dependence revealed these transitions to be partly spontaneous and partly collision-induced. Such delayed decompositions must result from energy storage in the $(N_2O)^+$ ion and transfer to a decomposition mode subsequent to acceleration of the parent ion.

CONTROLLED THERMONUCLEAR RESEARCH

DCX-1 Facility. – The energy-loss experiments with the neutral-particle spectrometer have been continued, with results that support the general conclusions of the preliminary work previously reported. In addition, however, it has been found that, as the trapped proton density is increased, the energy distribution of the escaping neutrals becomes increasingly broader. This spreading phenomenon is characteristic of both gas and (carbon) arc breakup, and radial and longitudinal spectrometer scans show that it is a gross characteristic of the trapped plasma. Attempts to correlate the phenomenon with the plasma radio-frequency spectrum have shown, in addition to the ion cyclotron frequency and its numerous harmonics, strong radiation at a number of very narrow frequency bands. These frequencies do not shift with changes in plasma density. Several experiments are planned for determining the origin of this radiation.

DCX-EPA. – This facility is still in the process of being moved to and assembled in Building 9201-2.

Ion Source Development. – Tests have continued on the 600-kv beam test facility. In an atmosphere of mixed Freon and nitrogen the tube has withstood 600 kv. A beam in excess of 50 ma has been obtained at 400 kv and has been focused with the magnetic solenoid lens to a small spot $\approx$ 40 in. below the lens.

Since a solenoid lens has focal properties proportional to the mass of the beam particles, it may be used for beam analysis. A lens of the type

used on the 600-kv beam test facility is being designed for use with the 100-kv test facility. With this lens it will be possible to study mass ratios from ion sources at currents up to 500 ma.

A duo-plasmatron ion source with an increased cup area in the form of a slit is being designed and constructed to determine whether currents in excess of an ampere can be accelerated and handled.

Up to 0.6 ma of analyzed H_2^+ has been obtained from the portable 100-kv diagnostic ion beam source. It is expected that at least 0.1 to 0.2 ma of analyzed H_1^+, H_2^+, or H_3^+ will be available reliably in approximately one week.

Theory. – The implications of energy loss of hot ions to cold electrons flowing into the DCX-II have been studied. The result is a modification of the characteristic S curve which produces appreciably larger critical currents. On the other hand, there is a large initial gain in trapped density as the arc is shut off, owing to the fact that the breakup remains high while the losses decrease. The net result of these two effects is more favorable than original calculations.

BIOLOGY AND MEDICINE PROGRAM

RADIATION EFFECTS ON BIOLOGICAL SYSTEMS

Genetic and Cytogenetic Effects of Radiations. – Genetic mapping of the ad-3 region of the mold *Neurospora* is being continued. The use of "leaky" ad-3 mutants in allelic crosses has presented new problems since they can grow on adenine-deficient medium. Evidence has now been obtained that benzimidazole can be used to differentially inhibit the growth of adenine-requiring and adenine-independent progeny in such crosses, and the influence of the addition of benzimidazole to the plating medium is being investigated in detail.

The development of a complementation map to indicate the nature of the interaction patterns of ad-3 mutants in the heterokaryon tests is also being continued. Attention is being focused at present on mutants induced by nitrous acid, since in marked contrast with mutants induced by x rays or by ultraviolet radiation, over 80% of such mutants show allelic complementation and exhibit a wide variety of different types of interaction patterns. Present data show a minimum of 28 different subunits in the complementation map, and the interaction patterns show that the map is definitely nonlinear. Over 1000 mutants induced by nitrous acid have not as yet been tested, and a major effort is being made to analyze the remainder of this sample.

Effects of Radiation on Paramecium. – As a part of the continuing investigation of growth processes in single cells of *Paramecium aurelia*, studies have been made of the incorporation of tritiated cytidine into nuclear and cytoplasmic ribonucleic acid. Previous investigations in collaboration with T. O. Caspersson of Stockholm have shown that there is no net increase in nuclear RNA during the first half of the interdivision interval, whereas RNA, together with deoxyribonucleic acid and protein, doubles in amount during the second half. The present studies show that cytidine is incorporated

into nuclear RNA at nearly the same rate throughout the whole interval. Most of the label is in the nucleus if the paramecia are fixed immediately after a brief labeling period, but most of it is in the cytoplasm if they are incubated in unlabeled medium for about an hour before fixing. These results suggest that the apparent constancy of nuclear RNA during the first half of the interval is the result of a balance between the rates of synthesis in the nucleus and release to the cytoplasm.

Pathology and Physiology. – Delayed mortality in irradiated F_1 mice that have received parental bone marrow has been attributed to an immunological reaction of antibodies produced by grafted cells against host tissue. C57BL, 101, (C57BL × 101)F_1, and (C57BL × DBA/2)F_1 mice were used for further examination of this hypothesis by injection of parental bone marrow into irradiated F_1 mice and vice versa. Both supralethal (850–1050 r) and midlethal (500–850 r) doses of x rays were used to determine the optimal conditions for good long-term survival of recipients and for permanent acceptance of transplanted cells. Differences in hemoglobin were used to follow repopulation of donor erythrocytes in irradiated recipients. The results indicate that more of the 900-r x-irradiated (C57BL × 101)F_1 recipients survived and permanently retained the graft when injected with 75×10^6 C57BL nucleated bone marrow cells than when injected with 15×10^6 cells. Reversions occurred earlier, moreover, when the smaller number of cells was used. Reversions were not found in 900-r x-irradiated (C57BL × 101)F_1 mice when strain 101 bone marrow cells were administered. Reversions in lethally irradiated (C57BL × 101)F_1 mice given C57BL bone marrow may be caused by a microenvironmental advantage for host cells or by an ability of the host to react weakly against grafted cells. Permanent transplants were obtained in 850-r x-irradiated strain 101 mice injected with 75×10^6 (C57BL × 101)F_1 bone marrow cells; reversions occurred when 15×10^6 cells were injected. When strain 101 bone marrow was injected into (C57BL × 101)F_1 mice, complete repopulation occurred within 50 days; but at least 60 days was required with injections of C57BL marrow.

Biophysics: Ultraviolet Studies. – The action spectrum for photoprotection from killing in the bacterium *Escherichia coli* B is being determined. The effective wavelengths lie in the range 3100 to 3700 A, with a single peak at 3400 A. The lower end of this spectrum is similar to that for photoreactivation, but the upper end lies at much lower values, suggesting that photoprotection either uses a different chromophore or uses the same chromophore less efficiently.

Studies of the synergistic action of x rays and ultraviolet radiation on chromosome breakage in *Tradescantia* pollen have been continued along several different lines of approach. Preliminary experiments with irradiation by fast neutrons instead of x rays showed an apparent decrease in the effect. These studies are being extended to alpha-particle irradiations. During the past month a large variation in the magnitude of the synergism has been encountered. It appears that this variation is a function of the degree of humidity and the amount of solar radiation received by the pollen before the laboratory treatments.

Since the synergism in the combined action of x rays and ultraviolet radiation on *Tradescantia* pollen is subject to considerable variation in magnitude from week to week, the factors governing the sensitivity of pollen to ultraviolet radiation alone are being investigated. The parameters under

study are temperature, humidity, and the amounts of near-infrared, visible, and near-ultraviolet radiation the material receives before exposure to the far ultraviolet (i.e., wavelengths in the 2500-A region). Although both humidity and temperature changes do affect the sensitivity of the pollen to ultraviolet, they do not appear to have a marked effect upon the synergism.

COMBATING RADIATION DETRIMENTAL EFFECTS

Radiation Protection: Living Cells. – Several systems are now established that make it possible to vary the radiosensitivity of bacterial cells. The largest change so far studied is one induced by control of the growth medium, and is dependent on the stage of the growth cycle, the presence of certain amino acids, the final pH of the culture, and, to some extent, the energy source. The amount of deoxyribonucleic acid, ribonucleic acid, and protein per cell correlates well with the average cell volume. Although resistant cells have about twice the cell volume of sensitive cells, and although the relative resistance to inactivation by x rays, ultraviolet, and heat increases with increasing cell volume, the relative resistance increases much more rapidly than does the cell size or the amount of the intracellular components measured. The expression of radioresistance can be altered by several pre- and postirradiation conditions, and these conditions are being simplified in order to pinpoint the cellular activity which is best correlated with development, loss, or expression of resistance.

Mammalian Radiation Recovery. – Experiments on mixed agglutination with red blood cells have shown that the ABO blood group antigens are present on the surfaces of tissue-cultured diploid human cells. Preliminary experiments with fluorescent antibody systems have further shown that the ABO blood group phenotype of a single cultured cell may be accurately determined. By use of the fluorescence method, clones of apparent type A cells have been detected in x-rayed mass cultures of type O cells.

Leukemoid leukocytes from F_1 hybrids caused some recovery from lethal irradiation injury in parent-type mice. Transplantation of F_1 hematopoietic cells was demonstrated in the parent-type recipients that survived. It is rare to fine leukemoid leukocytes causing recovery from radiation injury when the donor and the recipient animal are not genetically identical.

Irradiated mice that have been kept alive with foreign adult bone marrow usually show permanent gross damage to their lymphatic tissues, but some animals are now being studied who show a delayed regeneration of their lymphatic system. In these the new lymphatic system comes from the donor bone marrow.

Radiation Immunology. – The production of antibodies (agglutinins and precipitins) by normal and sensitized spleen cells is being studied by immunochemical techniques in diffusion chambers implanted in the peritoneal cavities of mice and rabbits. When preimmunized rabbit spleen cells were incubated with bovine serum albumin, antibody-containing cells could be demonstrated on the third day ($\sim$1% of the population), reached a peak of $\sim$6% on the fifth to seventh day, and decreased to $\sim$1% on the 15th day. Agglutinins (against sheep red blood cells) were detected in chambers

containing nonprimed homologous and heterologous (rat) spleen cells and antigen. Antibody was detected on the fifth day (titer 1:4), and at 12 days the titer was 1:64. No titratable antibody was demonstrated in the serum of the recipient at these times.

The in vivo tissue culture model offers a rational approach for determining whether a potential antibody-forming cell can make a simultaneous response to two or more antigens. Validity tests, in which given numbers of spleen cells from donors immunized against different antigens (A and B) were injected simultaneously into the irradiated recipient, have been carried out. The results demonstrate that, in response to either or both of the specific antigens, the cells respond to their respective antigen without interference or cross reaction.

Experiments with the model for the study of homologous transplantation antigens showed that the antigenic potency of viable cells was considerably higher than that of dead cells (100-fold). Persistence of viable cells, resulting in prolonged antigenic stimulus, was postulated as contributing to this phenomenon. Donor cells have now been labeled with H_3-thymidine, and the percentage of labeled cells in the spleen of the recipient was determined at various intervals after injection. The results definitely showed a longer persistence of the viable cells.

TRAINING, EDUCATION, AND INFORMATION PROGRAM

International Reactor Program at ORNL. – The ten Hazards Evaluation students of the 1959–60 session of ORSORT have completed nine weeks of a study project which consists in the preparation by the student of a report covering a specific problem pertaining to one of three different types of reactors. This program will terminate October 28, 1960.

The committee on admissions met in August to select qualified applicants for the third session of foreign programs, starting November 14, 1960. Thirty students, representing ten different countries, were selected and have been notified of their acceptance by the Division of International Affairs, AEC.

Applicants selected for the 1960–61 session of ORSORT are as follows:

Program	Number of Students
Hazards Evaluation	10
Operations Supervision	20
AEC trainees	15
TVA trainees	6
Carbide ORSORT (full or half time)	5
Carbide ORSORT (part time)	38
Total	94

At present, 54 students are participating in the pre-ORSORT program, which ends the second week in November, when regular ORSORT courses begin.

ASEE-AEC Summer Institute. — Twenty-one participants in the 1960 ASEE-AEC summer institute, conducted by North Carolina State University, concluded the last week of their program with experiments at ORSORT. The ORSORT work comprised seven experiments in radiochemistry and physics, including a criticality experiment at the Pool Critical Assembly; tours; lectures; and preparation of reports covering the experiments.

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OAK RIDGE NATIONAL LABORATORY

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