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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

NOVEMBER 1958

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

November 1958

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.

PROGRAM 4000 – REACTOR DEVELOPMENT

HOMOGENEOUS REACTOR PROJECT

Homogeneous Reactor Test. – Recovery of corrosion specimens from the core vessel, which began in October, was concluded. A total of 12 of the 22 specimens that became detached from the core corrosion assembly during run 17 was recovered. The remainder of the specimens were at inaccessible locations in the core diffuser-screen area.

As was reported last month, the major portions of the first, second, and fifth screens from the top are detached from the core vessel. The first and second screens were lifted and held in a vertical position. The locations where the first and second screens were formerly attached were studied through a tilting-mirror periscope. In most cases it appeared that the area of contact had been broken at the wall, although in some instances screen nubs were seen attached to the wall. Areas of localized attack were apparent on the vessel wall at several locations.

The screens were replaced in their normal positions after retrieving and viewing operations in the core were completed. No attempt was made to permanently attach the loose screens.

The installation of a new entrance line for the chemical processing plant was completed. The intake of the line will be at the eastern part of the core vessel wall near the top diffuser screen and may make it possible to remove separated phases to the chemical plant for further study.

In run 18, now commencing, the investigation of fuel stability will continue. (AEC Activity 4103.3)

Fuel Processing. – Further tests of the mercury-cathode electrolytic method for removing nickel from HRT fuel solutions showed that the nickel content can be

reduced to 5 to 10 mg per kg of D_2O. Preliminary data indicate that manganese can be controlled to about the same level. Analyses for mercury in the fuel solutions after electrolysis showed the quantity of mercury to be below the lower limit of detection, 10 ppm. Uranium losses to the cathode have been less than 0.03%. When a stainless steel cathode was used, the removal of nickel and manganese was less complete.

Tests of the UO_4 precipitation method for decontaminating HRT fuel solution showed that essentially the same decontamination was achieved as with simulated fuel solutions. The decontamination factors obtained in a single precipitation were: Cu, 86; Ni, 14; gross beta, 5; Sr, 151; Ba, 72; Ce, 2.2; total rare earths, 11; Cs, 7; and Ru, 6.

Visual observations of simulated HRT fuel solution sealed in quartz tubes with corrosion-product solids or metal specimens showed that such contaminants do not lower the temperature at which a second phase appears by more than 2 or 3°C. (AEC Activity 4103.1)

Gaseous Fission Product Disposal. — Activated charcoal did not catalyze the recombination of hydrogen and oxygen at temperatures up to 200°C. Oxidized steel wool, however, appreciably catalyzed the reaction at 200°C.

Propagation of the explosion front in a hydrogen-oxygen mixture through tubes filled with charcoal was rapid and complete. With a stoichiometric mixture of hydrogen and oxygen, a hydrogen-oxygen explosion front passed through the length of a 28-ft charcoal trap. This indicates that the combustion of the charcoal in bed A during the startup for HRT run 17 may have been initiated by deuterium-oxygen flame propagation from the recombiner to the point of ignition of the charcoal. (AEC Activity 4103.1)

Blanket Processing. — Equipment alterations and use of a CH_3OH-H_2O mixture as a solvent for the thorium nitrate feed to a fluidized bed of ThO_2 improved operation and permitted continuous denitration for up to 10 hr. However, small new particles of ThO_2 were produced instead of growth on the initial ThO_2 particles. The performance previously calculated for a slurry-loop fill system using hydroclones was demonstrated experimentally at room temperature. Fabrication of a 3.5-in.-dia hydroclone to test removal of over 5-μ-dia corrosion-product particles as a processing method was completed and given a successful preliminary test. Flame denitration produced toroid test quantities of mixed thorium-uranium oxides at 1200, 1400, and 1600°C in a reflected flame. The first toroid test of 1550°C material indicated no degradation. (AEC Activity 4103.1)

Thorium Oxide Slurry Development: Oxide Preparation. — One ton of 800°C-fired thorium oxide of 2 1/2-μ nominal average particle size was prepared from thorium oxalate which had been precipitated at 30°C and digested for 48 hr at 85°C. About 600 lb of 1600°C-fired thorium oxide was classified to remove particles >5 μ.

A technique was developed for coprecipitating uranous oxalate and thorium oxalate at U/Th ratios of up to 0.1 and possibly up to 0.25. Kilogram quantities of thorium-uranium oxide with U/Th ratios of 0.05, 0.08, and 0.10 were prepared by co-precipitation of the oxalates and subsequent thermal decomposition for evaluation in toroid tests. (AEC Activity 4103.1)

Thorium Oxide Slurry Development: Slurry Irradiations. — A slurry of 1000°C-fired thorium-uranium oxides (2.8% U^{235}) irradiated in the ORNL Graphite Reactor under an overpressure of D_2 gas with no recombination catalyst showed much lower equilibrium gas pressures than a similar system under air. This could be attributed to a lower G value (<0.6 mole of D_2 per 100 ev) for radiolytic-gas production or to an accelerated pickup of catalytically active (i.e., for gas recombination) corrosion products.

A slurry (500 g of Th per kg of D_2O) of thorium-uranium oxide solids (3% natural uranium) containing a palladium oxide catalyst (122 ppm; based on slurry solids) was obtained from the Westinghouse pump loop and, after being spiked with enriched uranium ($U^{235}/Th = 0.005$), was irradiated in the LITR. No radiolytic-gas pressure was observed during a two-week irradiation period.

Samples of a D_2O slurry of 1600°C-fired oxide (500 g of Th per kg of D_2O) containing ~ 0.01 m MoO_3 catalyst were circulated in a high-temperature loop under O_2, then D_2, and finally O_2 overpressures. Pumping under O_2 initially for 187 hr decreased the catalytic activity (after an induction period) from 3.7 moles of D_2 per hour per liter at 280°C and P_{D_2} = 100 psi to 2.0 moles of D_2 per hour per liter under the same conditions. The use of a D_2 atmosphere in a 307-hr test removed the induction period and increased the rate to 7.3 moles of D_2 per hour per liter at 214°C and P_{D_2} = 100 psi. A second pumping under O_2 for 287 hr did not reduce the catalytic activity of the slurry for gas recombination. (AEC Activity 4103.1)

Mathematics and Computation: Homogeneous Reactor Development. — The age of fission neutrons in D_2O-H_2O mixtures was correlated in terms of an age-diffusion model. For 99.8% D_2O, the age to indium-resonance energy was 109 cm^2, with the Gaussian age contributing 51.8 cm^2.

A number of machine programs were written; one calculates the reactivity changes associated with slurry settling in one-region aqueous thoria reactors. The harmonics method for calculating reactor criticality was programed; criticality is based on the vanishing of a determinant of the seventh order. A bare-reactor survey calculation has been programed in which resonance fissions are considered; effective resonance integrals are assumed to be a function of fuel concentration. In addition to critical fuel concentration, reactivity coefficients and conversion ratios are obtained.

Two-group nuclear constants have been prepared for thoria-H_2O-D_2O slurries for temperatures of 20, 100, and 200°C. (AEC Activity 4103.1)

Reactor Analysis. — Power excursions and oscillations observed during HRT operation were analyzed. "Excursions" are associated with relative power changes greater than 7%; "oscillations" are associated with "normal" power fluctuations of of 1 to 2%. The frequency of power-fall excursions had little correlation with power level and was much lower than that of power-rise excursions. In run 17 there were four excursions in which the power increased more than 100%; the largest percentage power increase in run 16 was 50%, while in run 14 the corresponding value was 20%. The frequency of excursions was between 1 and 2 per hour. The frequency of the oscillations was about 13 per minute at a reactor power of 3.5 Mw.

Parameter studies of the nuclear characteristics of single-region slurry reactors were extended to include moderator mixtures of light and heavy water. At 280°C, with a thorium concentration of 200 g/liter, a 4-ft reactor containing water of 95% D_2O content had the same critical fuel concentration as a 4.5-ft reactor containing 99.8% D_2O. At 200°C the reactor diameter had to be greater than 1.5 ft for critical-mass ratios (ratio of U^{235}/Th) less than 0.10.

The nuclear characteristics of reflected slurry reactors were obtained; average temperatures considered were 20, 100, and 200°C, while the moderator consisted of various mixtures of H_2O and D_2O. With core thorium concentrations between 200 and 400 g/liter in 2- and 2.5-ft-dia cores, the lowest critical-mass ratio occurred when the water contained 60 to 80% D_2O. The reactivity worth of the reflector increased with decreasing core diameter. With an initial reactor temperature of 100°C and with a stainless steel core wall 3/8 in. thick, the reflector of a 1-ft-dia core was worth about 22% Δk_e; that of a 2-ft-dia core was worth about 7% Δk_e. (AEC Activity 4103.1)

Homogeneous Reactor Instrumentation. — An experimental differential-pressure transmitter designed so that the sensing diaphragm will be flush with the process-vessel wall to avoid line plugging in slurry applications is being fabricated by The Foxboro Company. The instrument, which is based on an ORNL conceptual design, is specified for a differential range of -100 to 0 to +100 in. of water at a pressure of 2000 psi. Higher differential ranges will be achieved by substituting more rigid range-determining springs. The instrument is designed for operation on processes with maximum temperatures of 300°C.

A gamma-sensitive ionization chamber, which utilizes alumina insulators to permit operation in high-temperature environments, was fabricated by Reuter-Stokes Electronic Components to meet an ORNL specification. The stainless steel chamber housing is seal-welded and filled with pure nitrogen at 10 atm pressure. The chamber, 8 in. long and 2 in. in diameter, is rated for operation in ambient temperatures to 400°C, although the connectors presently used would limit the operation to lower temperatures. (AEC Activity 4103.1)

HRP Design. — Preliminary cost estimates for two concepts of a replacement pressure-vessel assembly for the HRT indicate a price of $500,000 for the nonremovable-core design and $575,000 for the removable-core assembly.

The tube for removal of a solution sample from the HRT core during reactor operation was redesigned so that a swivel joint permits positioning of the intake end near the intersection of the top screen and the core wall.

A device was designed for measuring the core wall thickness at any point from the uppermost diffuser screen to the circumferential weld at the top of the vessel.

Special tools were designed for use during the reactor shutdown operations. These include grippers for specimen removal, a cutting device for removing samples of the screens, and equipment for projecting external light into the reactor core for viewing operations. (AEC Activity 4103.1)

HRP Metallurgy. — A microexamination of the Zircaloy-2 screen samples removed from the HRT after run 17 revealed a single area near the center of the top screen that had been heated well into the $\alpha + \beta$ region. The transformed area extended around the circumference of a single hole and in one area extended through the screen thickness. If heating took place in other areas, it was not sufficient to cause recrystallization of the cold-worked structure. With short heating times such recrystallization should have been evident at temperatures of 700°C or higher. All the screens contained a large number of sheet-type intermetallic stringers and shear cracks around most of the holes. In many cases the two defects were combined. During operation the majority of the cracks had been corroded and filled with tightly packed oxides. Many of the cracks had propagated across an entire web, resulting in flaking of layers of metal from the surface.

Isotopic separation of removed gas is being used to assist in separating deuterium, picked up by zirconium during in-pile exposure, from hydrogen, picked up in handling and fabrication. It has been shown that no deuterium was picked up by Zircaloy-3A or zirconium-niobium samples exposed in the core of in-pile loop L-4-13 nor by Zircaloy-2 exposed in the HRT blanket during run 13. Deuterium, in appreciable quantities, was picked up by the top screen sample; smaller but constant amounts were picked up by the next three screens.

As a method of simplifying fabrication and eliminating welds in the next core tank, Zircaloy-2 plate, 5/16 and 1/4 in. thick, was successfully spun into 15-in. (half-size) hemispheres. It appears that spinning will yield hemispheres meeting the diametrical and thickness tolerances with only a minimum of machining or pressing to final size. (AEC Activity 4103.1)

Laboratory Corrosion Studies. — Stressed specimens of cast type 347 stainless steel have shown complete immunity to stress-corrosion cracking in a number of environments that normally cause cracking of wrought austenitic stainless steels. The environments examined include: (1) boiling 42% $MgCl_2$ solution, (2) boiling and aerated 0.04 m UO_2SO_4–0.02 m H_2SO_4–0.005 m $CuSO_4$ solution containing 50 ppm of chloride, and (3) water at 300°C containing 100 ppm of chloride and

approximately 10 ppm of oxygen. Although no cracking was observed, some subsurface attack was noted in the first two environments.

Nitrided titanium-55A, titanium-75A, and Zircaloy-2 specimens were exposed in oxygenated 0.04 m UO_2SO_4–0.025 m H_2SO_4–0.01 m $CuSO_4$ solution and in deionized water at 300°C. All titanium specimens corroded at rates less than 0.2 mil/year in both environments; Zircaloy-2 corroded at a rate of 14 mils/year in each environment. The nitriding process produced substantial surface cracking in all materials and a pronounced volume growth in the Zircaloy-2; therefore it is doubtful that the process can be used successfully for reactor applications. (AEC Activity 4103.1)

Small-Scale Dynamic Slurry Corrosion Tests. — Circulation at 26 fps for 100 hr at 280°C of oxygenated slurries (1500 g of Th per kg of H_2O) of thoria prepared by the draft-tube precipitation method indicated no correlation with calcination temperature or average diameter, the relative aggressiveness being expressed as the following multiplicative factors: batch DT-5 (1600°C, 2.1 μ), 1.6; batch DT-5, sized (1600°C, 3.1 μ), 1.0; batch DT-2-3 (1600°C, 3.8 μ), 13; batch DT-4 (650°C, 1.1 μ), 5. Relative susceptibility to attack of different specimens, expressed as multiplicative factors, was: type 347 stainless steel, 1.0; type 347 stainless steel toroid (different flow), 0.18; SA-212-B carbon steel, 1.2; titanium, 0.24; Zircaloy-2, 0.06. The normalized attack rate for type 347 stainless steel with sized batch DT-5 of 24 mils/year slightly exceeds usual values but is at least partially attributed to the shorter run at a higher concentration.

The aggressivensss of aqueous slurries of 1600°C-calcined thoria microcubes circulated at 26 fps for 100 hr at 280°C at concentrations of 250, 1000, and 1500 g of Th per kg of H_2O under oxygen (seven tests) and hydrogen (one test) atmospheres increased by a factor of 1.6 as average particle diameter increased from 2.1 μ to 3.1 μ. Rates under hydrogen averaged 30 to 40 times greater than those under oxygen, increasing for all materials. The normalized rate for type 347 stainless steel in an oxygenated 2.1-μ thoria slurry at a concentration of 1000 g of Th per kg of H_2O was 10 mils/year. Relative to this, multiplicative factors for other materials were: type 347 stainless steel toroid (different flow), 0.2; SA-212-B steel, 4.4; titanium, 0.3; Zircaloy-2, 0.04. Multiplicative factors indicating the relative aggressiveness at different concentrations were: 250 g/kg, 0.23; 1000 g/kg, 1.0; 1500 g/kg, 0.68. (AEC Activity 4103.1)

Dynamic Slurry Loop Corrosion Tests. — The second and third operational phases of a loop evaluation test (total time, 1297 hr) of a slurry of thoria prepared for the ORNL-PAR (Westinghouse) in-pile loop experiment were completed. The slurry of 2.4-μ, 1600°C-calcined thoria was circulated at 280°C and at an average concentration of 446 g of Th per kg of D_2O; ~0.01 m MoO_3 was included in the slurry. The second phase consisted in 478 hr of operation with a D_2-A atmosphere; the third and final phase was for 335 hr with an O_2 atmosphere. No concentration loss (under O_2) was noted at 4 fps in the 1 1/2-in. loop piping, but significant

circulating-concentration lowering was noted at 2 fps. The yield stress of a slurry sample containing 500 g of Th per kg of D_2O after D_2 atmosphere operation was 0.6 psf; that of a similar sample after O_2 atmosphere operation was 0.1 psf.

Generalized corrosion rates for stainless steel components of the loop were: D_2 atmosphere, 6 mils/year; final O_2 atmosphere, 1 mil/year. The periphery of the pump impeller, of type 347 stainless steel, was attacked, but the titanium pump head was not. Over-all specimen attack rates, in mils per year, were:

Velocity	347 SS	Zircaloy-2	Inconel	Titanium	Croloys 2 1/4 and 5	430 SS
20 fps	0.3	0	3	<0.1	2	0.5
40 fps	26	1	8	0.6	4	4

In another 280°C loop test with oxygenated slurry (277 g of Th per kg of H_2O) prepared from 0.9-μ, 1600°C-calcined thoria, the attack rates of chromium-alloy steels ranging from ASTM-A-7 (no Cr) to type 347 stainless steel (19% Cr) at 22 fps increased from 0.1 to 4 mils/year as the chromium content diminished, following the relation

$$\log(\text{alloy rate}/347\text{ SS rate}) = -k\,(\%\ \text{Cr})^{1/2}.$$

(AEC Activity 4103.1)

In-Pile Autoclave Solution Corrosion Studies. — An autoclave which employed a solution of 0.17 M UO_2SO_4 and 0.04 M $CuSO_4$ in D_2O was operated above the two-liquid phase separation temperature (285°C) in beam hole HB-5 of the LITR. An exploratory out-of-pile test of the uranium-copper distribution in a similar solution, except with 0.011 M $CuSO_4$, at 307°C showed a copper-to-uranium concentration ratio of 0.04 in the heavy phase and 0.14 in the light phase, as compared with 0.06 in the original one-phase solution. In the out-of-pile test approximately equal total quantities of uranium were found in each phase, with the volume of the heavy phase about 2% of that of the light phase.

Under irradiation the corrosion rates as indicated by oxygen consumption were 10 to 20 mils/year at 270 to 320°C — rates which might have been expected in a one-phase solution. Postirradiation examination showed no visual evidence of preferential attack on specimens or bomb walls. A loose pin which lay on the bottom of the bomb during exposure and which should have contacted heavy phase, if it was present and accumulated, showed a weight loss per unit of time similar to that of the other specimens. Radiolytic-gas pressures at temperatures above the probable temperature of two-liquid-phase separation were in most cases abnormally high as compared with the radiolytic pressures at the lower temperatures. This behavior may have resulted from a change in the uranium and copper distribution in the two phases from that in the single-phase solution. (AEC Activity 4103.1)

HRP Service Corrosion. — Specimens exposed in the blanket region of the HRT during runs 13 and 14 were examined and weighed in the as-removed condition. In addition, scale analyses were performed on a portion of one specimen each of Zircaloy-2 core-tank material, type 347 stainless steel, and Ti-A40. Uranium was detected in the scale on the titanium only, in an amount equivalent to 2 $\mu g/cm^2$. The appearance and weight measurements of specimens indicate that attack was in no case appreciably different from that which would have occurred in the absence of radiation.

A new, freshly machined type 347 stainless steel flange with an attached stainless steel specimen was installed in the HRT following run 14 and was removed for examination following run 16. All surfaces that had been exposed to solution were covered with an off-white-colored scale, which easily flaked off, leaving a rust-red deposit next to the metal. Scale analyses showed a scale weight of about 6 mg/cm^2 and indicate that the scales consisted, primarily, of the oxides of the constituents of stainless steel and of ZrO_2. The metal lost by the defilmed stainless steel specimen corresponds to an average weight loss of 0.32 mg/cm^2 (difference between original and final weights); hence the oxidation products of the specimen did not contribute appreciably to the scale weight. It is likely that the major portion of each scale was formed by deposition from solution of oxides generated elsewhere in the reactor. The amount of zirconium oxide found is no more than that expected for moderate corrosion of the Zircaloy-2 core tank during run 16, with subsequent uniform deposition on surfaces outside the core. (AEC Activity 4103.1)

CIVILIAN-POWER ADVANCED GAS-COOLED REACTOR

Physics. — Multigroup diffusion calculations were performed for the Kaiser-ACF prototype reactor (GCPR) in both the radial and axial directions to determine capture gamma-ray source distributions for radiation heating and shielding calculations. The models employed included the graphite-support grid, the control-rod bank inserted to a depth of 4 ft, and part of the biological shield.

Neutron transport calculations made with the SNG code were performed for the reactor cell profile in slab, cylindrical, and spherical geometry. The calculations were successful in the slab case, but it was necessary to employ a new approximation in integrating the Boltzmann equation in the SNG code to obtain a converged solution for the cylindrical case. Presumably the new approximation will also yield satisfactory results for the spherical case. It should be possible now to produce large numbers of cell calculations on demand.

A two-group calculation of a homogeneous GCPR was performed in S_4 approximation with the constants used by ACF in their two-group diffusion-theory calculations. The group fluxes obtained by the two methods are indistinguishable throughout the core and the reflector. This does not prove that the calculated fluxes are correct, but only that the conditions of diffusion theory are satisfied in the problem. The S_4 results show the fluxes to be nearly isotropic, even close to the outer reflector boundary.

Radiation heating in the pressure vessel of the GCPR was calculated. By far the largest contribution is from gamma rays produced in the capture of thermal neutrons in the pressure vessel and in the thermal shield. The calculated result, corresponding to a surface heat flux of about 320 $Btu \cdot hr^{-1} \cdot ft^{-2}$, is about 60% of that quoted by Kaiser, but the result is directly proportional to the thermal-neutron flux in the pressure vessel and is therefore somewhat in doubt. If the heat load or the thermal stress proves to be uncomfortably large, the thermal-neutron flux can easily be reduced by borating the outer few inches of graphite or by some similar means. (AEC Activity 4110)

Fuel Element Fabrication and Inspection. — Solid pellets of normal and 3% enriched UO_2 were prepared for prototype capsule irradiation tests. The compacting die for preparing cored prototype-size pellets was received, and production experiments were initiated.

A review of the irradiation test capsule design and analytical evaluations of the internal pressures that may be developed by the fission gas revealed the need to provide a gas-expansion volume in the capsules that contain solid pellets. Such expansion volumes will not be needed in the capsules containing cored pellets.

The first ETR capsule assembly, which incorporates solid pellets, was completed. Some difficulty was encountered both with the final closure weld in the dry box and in accomplishing an acceptable leak test. These difficulties, however, were no more severe than would be expected for a first assembly, and it appears that later assemblies should not present serious problems.

Twenty-four end-cap closure specimens containing defects introduced by intentionally varying welding conditions were prepared for metallographic examination. The destructive test data thus obtained will be correlated with nondestructive test data and with the welding conditions. Another set of weld samples was encapsulated in quartz for aging studies at 1300 and 1500°F. Work continued in the study of welding variables in order to determine optimum welding conditions for the three basic capsule end closure designs.

In-plant nondestructive testing of ~1650 ft of 3/4-in.-OD, 0.020-in.-wall, type 304 stainless steel fuel capsule tubing fabricated at the Superior Tube Company for the critical experiment at Hanford was completed. About 400 ft of the tubing was rejected at the plant, and the remainder was sent to ORNL for further inspection. Of the first 1150 ft examined at ORNL by the eddy-current technique, 34 lengths displayed indications that exceeded the indication of the reference standard. The tubing seems to contain small, sharp, dimensional changes, discontinuities, and other anomalies. Ultrasonic inspection revealed a few very small discontinuities, but no gross defects were noted.

Means were developed for furnishing to the fabricator the enriched material for the UO_2 pellets required for the critical experiments to be performed at Hanford.

Arrangements were also made for in-plant inspection of the material. The 0.050-in.-thick enriched foil needed for the experiment is being fabricated by the ORNL Metallurgy Division. An order was placed for the 3 1/4-in.-OD, 2 3/4-in.-ID, 3-in.-long, boron carbide shapes needed for the control rods. The 1 1/2% -boron-containing type 304 stainless steel specified for the experiment is not available, but an alternate material containing 2% boron is available that will probably be satisfactory. The 850 MgO spacers required were prepared and shipped to Hanford.

The 250 normal UO_2 pellets needed for the ORNL out-of-pile forced-convection loop experiments were obtained on a purchase contract. The MgO spacers for these experiments will be supplied from the excess furnished to Hanford.

Nondestructive tests were made on approximately 120 ft of 1/2-in.-OD, 0.003-in.-wall, type 304 stainless steel tubing. Wall thickness measurements indicated a consistent variation from 0.0027 to 0.0031 in. The length, warped shape, and fragile structure of the material precluded inspection by the pulse-echo ultrasonic technique. A sample penetrant inspection revealed the presence of some localized pits on the outer surface. A sample inspection with low-energy x rays revealed a mild striated condition in the longitudinal direction which seemed to be thin wall areas too localized to be detected with the metal identification meter. In addition, a few discontinuities were noted by this inspection.

Cost information was assembled from suppliers of beryllium tubing of the types to be obtained for advanced materials research: (1) 50 ft of extruded seamless tubing, 0.750-in. ID, 0.040-in. wall, and (2) 50 ft of extruded and machined tubing of the same dimensions. Two price quotations, $7400 and $3950, were received for item 1 and none were received for item 2. (AEC Activity 4110)

Mechanical Property Tests of Capsule Tubing. — A buckling test of 0.015-in.-wall type 304 stainless steel tubing was discontinued at the end of 500 hr. The test was conducted at 1500°F with a 300-psi external pressure on specimens having various diametral clearances. Uniform collapse was obtained of those specimens with diametral gaps of 0.002 and 0.005 in., but the specimen with a 0.010-in. diametral clearance buckled in a two-lobe mode.

In an attempt to obtain uniform collapse of a tubular specimen having a 0.010-in. diametral clearance, a specimen was tested at 1700°F with an external pressure of 500 psi. Examination of the specimen at the end of 18 hr revealed that uniform collapse had occurred.

A test to investigate the possible rupture of the cladding material when small cracks or fissures are present in the UO_2 slugs was conducted on a specimen having a 0.010-in. diametral clearance between the inserts and the tube wall. Test conditions were the following: uniform temperature, 1300°F; hot-streak temperature, 1500°F; external pressure, 300 psig. The test was discontinued at the end of 500 hr. Little change was noted in the specimen, so it will be tested for an additional 1000 hr.

Tube burst tests performed on 0.020-in.-wall type 304 stainless steel tubing gave the following results:

Temperature (°F)	Internal Pressure (psig)	Time to Rupture (hr)
1500	300	1148
1500	400	375
1300	700	306

The end cap of the first specimen was fusion-welded, and the rupture occurred in the fusion zone of the weld. The end caps of the last two specimens were butt-welded. The failure in both these specimens occurred in the axial direction in the tubing. (AEC Activity 4110)

Materials Compatibility. — Metallographic examinations were completed of specimens from static pot No. 6, which contained helium, TSF graphite, and structural materials and which operated at 500°F for 100 hr and at 1250°F for 900 hr. The surfaces of the type 304, 310, 316, and 410 stainless steel specimens showed layers characteristic of decarburization. The layers varied in thickness from 0.1 to 0.5 mil. The type 304 stainless steel specimen also showed areas at the surface where ferrite had apparently formed, an effect which is also indicative of decarburization. The DB-2 alloy (7% Al–5% Cr–bal Fe), Croloy, and T-1 steel specimens showed no evidence of attack. The stainless steels showed little change in mechanical properties as a result of the test. However, the Croloy and T-1 steel specimens showed decreases of 50 to 90% in strength and increases of about 80% in ductility. These changes were probably due to annealing caused by the high operating temperature and the long time of the test.

Static pot test No. 7, in which the materials were beryllium, TSF graphite, and helium, was terminated after completing 100 hr at 500°F and 900 hr at 1400°F. The beryllium samples were grossly attacked. The heavy gray scale which formed on the specimens seemed to be crystalline and was not adherent to the surface. The stainless steel rods which supported the beryllium samples showed a loosely adherent gray scale which differed in appearance from the beryllium scale. It was difficult to determine by visual inspection whether this scale formed on the stainless steel or was deposited there. All samples were submitted for x-ray diffraction analysis.

Analyses made of gas samples taken during static pot test No. 7 are being evaluated. Plots of the total impurity concentration vs time for this test show no major differences from other tests at 1400°F in which stainless and low-alloy steel specimens were utilized. This suggests that the rate of reaction of the impurities with the beryllium, while initially quite fast, slowed down sufficiently for the loss of impurities from the helium atmosphere to be eventually controlled

by the stainless steel walls of the test vessel, which comprised a much larger surface area than that of the beryllium specimens.

Static pot test No. 8, in which the materials are TSF graphite and test spcimens of stainless and low-alloy steels, has completed 696 hr of a scheduled 900-hr program of operation at 1100°F. Impurity concentration levels measured in helium samples removed from this pot have remained at relatively high values, compared with those of similar tests at higher temperatures (1250° and 1400°F). The principal contaminants remaining are H_2, CH_4, and N_2 + CO. The total concentration of all impurities measured after 650 hr at 1100°F was 0.525%. The total concentration remaining in previous tests at 1400°F after a similar period of operation has been in the range 0.01 to 0.02%, and in a single test at 1250°F it was 0.0175%.

Thermal-convection loop No. 5, which contains TSF graphite, has completed 480 hr of a 900-hr test at 1300°F. Total contaminants in the helium atmosphere have now decreased to a few hundred parts per million. Equipment for test No. 6 is being assembled. AGOT graphite will be used in this test. Procurement and assembly work continued on both a high-pressure thermal-convection loop and a high-pressure forced-convection loop.

A study was made of the reaction between type 304 stainless steel and CO at 1500°F. Pure CO was generated by reacting degassed activated charcoal with purified CO_2 at 1800°F and approximately 1 atm under static conditions. The resulting CO was further purified in a reservoir containing Ascarite and Anhydrone to remove the last traces of CO_2 and H_2O. This prepared gas, at a pressure of 20.5 cm Hg, was allowed to react with a 0.002-in.-thick stainless steel specimen at 1500°F for 118 hr. The system pressure decreased rapidly under these conditions until the reaction chamber showed that a high vacuum was attained. The metal was covered with a greenish, adherent, and nonmagnetic oxide film. Posttest examinations show no carbon deposits. Carbon analyses made before and after testing showed that the metal was carburized and that apparently no carbon was present in the film. The reaction $14Cr + 3CO \longrightarrow Cr_2O_3 + 3Cr_4C$ is proposed as the mechanism of carburization of the steel. (AEC Activity 4110)

Radiation Effects Studies. — Analyses were completed on the fission gas samples obtained from the first two miniature UO_2–stainless steel capsules, which were irradiated in the LITR for about 12 weeks in a flux of 2×10^{13} neutrons·cm^{-2}·sec^{-1}. Gases from the top and bottom sections of capsule 1-2 showed slight differences that can be attributed to a difference in irradiation temperature. The analytical results are listed below:

	Volume Released (cm^3)		
	Capsule 1-1	Capsule 1-2	
		Top	Bottom
Kr^{88}		$4 \times 10^{-8} \pm 30\%$	$8 \times 10^{-8} \pm 30\%$
I^{131}	$3 \times 10^{-7} \pm 30\%$	$1.4 \times 10^{-8} \pm 30\%$	$2.5 \times 10^{-8} \pm 30\%$
Xe^{133}	$3 \times 10^{-5} \pm 10\%$	$2.0 \times 10^{-5} \pm 10\%$	$2.7 \times 10^{-5} \pm 10\%$
Xe^{135}	$3 \times 10^{-7} \pm 10\%$	$1.6 \times 10^{-6} \pm 10\%$	$2.3 \times 10^{-6} \pm 10\%$

Two SiC-Si coated graphite specimens, which were irradiated to a dose of 9×10^{19} neutrons/cm^2 (thermal) in the ORR at cooling-water temperatures, were heated at 1100°C for a total of 193 hr in a moving air stream. No significant changes were observed. The specimens were prepared for reinsertion in the ORR and will be tested again after a dose of 4×10^{20} neutrons/cm^2. Two additional specimens are being irradiated that will be tested after receiving a dose of 10^{21} neutrons/cm^2 (thermal).

Most of the equipment needed for hot-cell testing of irradiated thermocouples was assembled and was installed in the cell. Test runs of the equipment were made on unirradiated wires. An improved jig for remote welding of Chromel-Alumel thermocouples was found to be satisfactory.

Construction work is near completion on the closed-cycle loop for testing fuel-element materials under irradiation in the ORR (experiment B-9). Pressure-drop data were obtained in an out-of-pile test.

Design work was completed on equipment for studying instantaneous fission-gas release during irradiation in the ORR. The present schedule indicates that the installation should be complete by the end of December 1958. Studies are being made in an attempt to formulate an order-of-magnitude theoretical prediction of the effect of temperature, density, power, and time on fission-gas release from a ceramic body.

Design work on modifications of ORNL hot cell 6 was completed, and the contractor set March 15, 1959, as the estimated date for completion of the modifications. Design work was nearly completed on the ORR hot cell, and fabrication orders were issued for much of the special equipment.

Four capsules containing UO_2 pellets are presently being irradiated in the LITR at central pellet temperatures of approximately 2300°F and sheath temperatures of 1300°F. The central thermocouples in two of these capsules have failed. The search for suitable thermocouple sheath materials for measurement of the high central pellet temperatures is continuing.

Installation of equipment for irradiation tests in the ETR is somewhat ahead of that for the tests in the ORR. A recent decision by the safeguard committee may cause some delay in the start of tests, however, inasmuch as the design of the facility will have to be modified to incorporate the secondary bulkhead requested by the committee. Components are being procured, and construction is under way on the loop to be installed in the G-E test reactor (GETR) at Vallecitos, California. (AEC Activity 4110)

Graphite Degassing Studies. — A system was constructed for studying the evolution of gas from graphite at high temperatures. Pending the completion of work on an induction heater, preliminary tests were made with a specimen of TSF

graphite at 200, 400, 600, 800, and 1000°C. The collected gases were submitted for analysis by mass spectrometer, but the results are not yet available. After minor modifications were made to improve the leak-tightness of the system, a specimen of AGOT graphite and a specimen of graphite from the Great Lakes Carbon Company were also degassed. Higher-temperature tests will be initiated upon completion of the induction heater. (AEC Activity 4110)

Heat Transfer Experiments. — An apparatus was assembled with which the bending of the rods in a heated fuel cluster could be observed optically and photographically and in which the Reynolds modulus of air flow around the heated tube could be varied. Visual observation indicated that 0.75-in.-dia rods spaced on 1.09-in. centers in a 3.18-in. channel and heated to 1000°F bowed outward at a Reynolds modulus of 75,000. Optical distortion in the Vycor channel wall prevented quantitative evaluation of the observed rod bending, but an attempt will be made to estimate the extent of the deflection from comparative photographs.

The design of an apparatus for determining fuel-element hot-spot location and magnitude was completed. The fuel-element cluster will be heated by electrical resistance, and it will be installed in a graphite channel. An apparatus for testing the thermocouple devices to be used in these tests was constructed. A number of thermocouple configurations were investigated for surface temperature measurements, and two were found to be satisfactory: (1) a small bead thermocouple held against the surface by a spring and (2) two spring-loaded thermocouple wires contacting the surface so that the tube wall forms part of the junction. (AEC Activity 4110)

Design Study of a Contaminated-Coolant Gas-Cooled Reactor (HGCR-1). — The proposed layout of equipment within the containment vessel of the HGCR-1 was altered to reflect the lower cost of external vs internal headers on the steam generators. Further, shielding was added between the external header and the steam generator to make possible direct maintenance on the header. Estimates were made of fuel fabrication and plant costs for the HGCR-1. The fuel fabrication cost for extruded elements was $36.80 per kilogram of uranium, and the plant cost was less than $250 per kilowatt of electrical output.

Data calculated from equations describing the release of activity from graphite-UO_2 fuel permitted the calculation of the activity in the gas and the amount of shielding required. These studies indicate that the graphite-UO_2 fuel material is quite effective in retaining fission products. Probably the most important function of the graphite, however, is delay of the release of activity until the nuclides have decayed into less energetic gamma-ray emitters. With the fuel temperature expected in the HGCR, the activity release is only ~0.15% of the activity generated inside the fuel or, for this case, an activity of 6×10^6 curies of gamma-ray emitters with energies greater than 0.5 Mev.

With this activity release, 3 ft of concrete is necessary to maintain a dose rate of 7.5 mr/hr adjacent to the steam generators during operation. The dose rate at

this location during operation is due to many short-lived nuclides as well as Y^{90}, Ba^{137m}, and La^{140}. One day after shutdown the dose rate is due primarily to La^{140}, and this activity remains as the main source until about 100 days, when Y^{90} and Ba^{137m} become predominant. At about 100 days the system activity has decreased sufficiently that maintenance can probably be carried out with no shielding at dose rates less than ~200 mr/hr.

The possibility of removing the longer-lived nuclides by using purification systems, and the further possibility of reducing the dose rate by decontamination of equipment, makes it probable that major maintenance on system components may be done directly or with portable shields by waiting for periods of 10 to 100 days. (AEC Activity 4110)

Applied Mechanics: Design Stress Analyses. — The equations for analyzing a grid structure, such as the diagrid used for supporting the core of the Calder Hall reactor, were derived. These equations include only the bending rigidity of the beams; the added influence of the torsional rigidity, which is small compared with that of bending, is also being examined. (AEC Activity 4110)

Thermodynamic Studies of Fuel-Element Shapes. — Preliminary heat transfer calculations for a flat-plate fuel element were completed. The design chosen for the study consisted of three UO_2 plates, 0.3 by 2.75 by 24 in., clad with 0.020-in.-thick stainless steel, placed parallel in a 3-in.-square coolant channel. The calculations were made for a 20-ft active channel length with the same power output as that of the central channel of the GCR-2 for the following conditions: coolant pressure, 300 psi; gas inlet temperature, 450°F; gas exit temperature, 1000°F.

The results indicate that the maximum temperature variation between the two sides of the outside fuel plates is 27°F and that it occurs approximately halfway through the channel. The maximum fuel plate surface temperature is 1211°F, and the maximum coolant temperature variation is 37°F. The friction head loss through the channel is 4800 ft of helium. The temperature variation on the outside plates is minimized by orienting each 24-in. fuel-element assembly 90° away from the preceding one and by selecting the optimum spacing between the plates. It is assumed that there is complete mixing of the coolant between each 24-in. fuel-element section.

Calculations of the temperature structure of multirod elements in a fluted channel were completed. The results are being analyzed and compared with those obtained for a circular channel.

Temperature profiles in the thermal shield and pressure vessel sections adjacent to the core were calculated from heat generation rates obtained previously. The temperature difference between the inside and outside surfaces of the pressure vessel due to internal heat generation was of the order of 2°F. (AEC Activity 4110)

Compressor Development. – Fabrication work is under way at the A-Tool and Gage Company, Detroit, Michigan, on a grease-lubricated compressor for the ORR gas-cooled loop for testing full-size fuel elements. A purchase requisition was issued for procurement of a gas-bearing centrifugal compressor from Armstrong-Siddeley Motors, Ltd., Conventry, England. Fabrication work continued at the AiResearch Mfg. Co. on the compressor required for an out-of-pile loop in which argon will be circulated. (AEC Activity 4110)

Design Review and Power Plant Analyses. – Kaiser Engineers and ACF drawings, outline specifications, and job reports were reviewed. A discussion was held with members of the Power Operations Group in the Chattanooga office of TVA concerning operation of electrical generating plants and network systems. A method for obtaining automatic adjustable-base-load operation from a generating plant was outlined. The equipment necessary is standard, and the cost of such equipment would be very low. The installation of such equipment on GCPR would permit the plant to operate automatically at constant power, independent of network frequency deviations. (AEC Activity 4110)

GENERAL REACTOR RESEARCH

Radiation Detector Development. – Two large NaI(Tl) crystals, comparable in size and geometry to the one studied at the Bulk Shielding Facility, were obtained on a loan basis from the manufacturer (Harshaw Chemical Company, Cleveland, Ohio). The responses of these crystals to various gamma-ray energies were determined by collimating the gamma rays into both the conical end and the cylindrical side of the crystal. The results indicated that these crystals respond in the same manner as other crystals with this geometry. A double peak for each gamma-ray energy was found when the gamma rays were collimated into the conical end. With the gamma rays collimated into the side of the cylindrical section, the response was more nearly Gaussian, with only one peak for each energy. It is believed that these studies support the theory that the double peaks are due to the unusual combination, for scintillation crystals, of a cylindrical and conical geometry. (AEC Activity 4202)

Liquid Metals Research. – The rate of mass transfer of copper in liquid mercury was studied over the temperature range 86 to 193°C in a Pyrex seesaw rig as a function of the relative hot-zone and cold-zone contact times, the rocking frequency, and the temperature difference between the hot and cold zones. The results obtained indicate that the rate of mass transfer increases with increasing hot-zone temperature, with increasing temperature difference between the hot and cold zones, and with increasing ratio of hot-zone contact time to cold-zone contact time. It may be concluded from these data that the mass transfer of copper in mercury under these conditions is probably controlled by the rate of solution in the hot zone. However, this interpretation may be doubtful, since varying amounts of undesired oxidation of the copper were observed during the tests.

A Pyrex loop was built with which solution rate constants can be measured for solids in liquid mercury. The rate of solution of nickel in mercury was measured under a single set of conditions.

An apparatus for measuring diffusion coefficients in liquid metals at temperatures up to 800°C was constructed. Measurements of the diffusion coefficients of silver in liquid lead-silver alloys were commenced. (AEC Activity 4203)

Fuel Element Catastrophe Studies. – A re-evaluation of the instantaneous release of fission products from melting STR-type Zircaloy fuel plate was completed, encapsulated disk-type punchings being used in order to avoid an ambiguous effect of an exposed fuel edge. Direct-coupling induction-type melting was used, and a series of melts was obtained both in air and in steam. The results for melting in air indicate quantitative loss of rare gases and 25 and 12% loss of iodine and cesium; no other fission products were observed to vaporize. Melting in steam was shown to cause only a slight increase in the release of iodine and of cesium, to 33 and 16%, respectively. As before, the rare gases are quantitatively released. Further examination of the vaporized products revealed about 3% of the strontium, about 1% of the barium, and less than 0.1% of the cerium. (AEC Activity 4252)

Reactor Safety Studies. – The first group of experiments in the study of the effects of voids in reflectors was completed with an approximately 4 x 7 element Pool Critical Assembly loading containing ~2% excess reactivity. The experiments included determinations of (1) the effect of centrally located voids, (2) the effect of varying the location of the void, and (3) the superposition of reactivity effects of several voids. Preparations are now being made for the second group of experiments, which will use a PCA loading containing ~3.5% excess reactivity.

Fabrication of the prototype control rod for the stainless-steel-clad BSR-II is nearing completion and soon will be life-tested. Fabrication of all fuel elements, standard and experimental, has been scheduled. Discussions were held at ORNL with SPERT-I personnel, and the essentials of a mutually acceptable test program were outlined. Testing at NRTS was scheduled for June 1, 1959. Preliminary estimates indicate that the maximum Δk/sec obtainable by controlled fuel plate insertion is insufficient for all the BSR-II tests in the SPERT-I facility. The use of a double-ended rod will be considered. (AEC Activity 4252)

Power Reactor Fuel Reprocessing: Darex Process. – Experiments showed that passivation of stainless steel fuel elements may be induced by use of insufficient Darex dissolvent; such passivation may be broken by the introduction of a trace of metallic copper into the dissolver.

Pulsed-column tests of a Darex process flowsheet based on 2.5% tributyl phosphate gave uranium losses of less than 0.001% at total nitrate salting strengths of about 6 M. The heights of the extraction and stripping stages were about 3 and 2 ft, respectively.

The Hanford assistance effort was started on the demonstration of a Redox first-cycle flowsheet in Building 3503. Preliminary runs with both standard and synthetic Darex-Redox feed solutions were made in a 2-in. glass column packed with Raschig rings. The standard feed flooded at 590 $gal \cdot hr^{-1} \cdot ft^{-2}$; the Darex feed flooded, in two tests, at 560 and 640 $gal \cdot hr^{-1} \cdot ft^{-2}$. The values of the height equivalent to a theoretical stage for standard and Darex feed were 6 and 12 ft, respectively.

Subsequent runs in the Interim-23 stainless steel equipment were also begun with Hanford-supplied Darex feed to which irradiated uranium had been added. Batch scrubbing tests on the HAP stream from Redox column runs indicated that a second cycle would give gamma decontamination factors of from 1 to 4; Ru^{106} was essentially the only fission product species present. (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Hermex Process. — The solubility of thorium in mercury containing 0.005 wt % uranium is 0.0018 and 0.0282 wt % at 50 and 356°C, respectively, the same as the corrected solubility in mercury alone. This is the concentration of uranium that would be introduced into the mercury in processing thorium containing 4000 g of uranium per ton. Although the uranium concentration in the mercury phase was expected to be 0.005 wt % over the range 356 to 125°C, it was actually observed to decrease from 0.003 wt % at 356°C to 0.00005 wt % at 125°C. The decrease in solubility is believed to be due to coprecipitation of UHg_4 with $ThHg_3$ at the lower temperatures. (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Pilot Plant Modifications. — Decontamination and modification of Building 3019 continued during this report period. The radiation backgrounds in all cell cubicles were reduced to less than 100 mr/hr. Plant modifications to provide facilities for Systems for Nuclear Auxiliary Power (SNAP) fuel element dissolution, codecontamination, and solvent treatment with a capacity of 400 kg of uranium per day were 90% completed.

Process modifications continued in Building 3505 with the objective of improving equipment reliability and on-stream time. When modifications are complete, this facility will include (1) a fuel element dissolver for low-level fuel, (2) a partitioning cycle for separating uranium and plutonium previously processed in the Building 3019 codecontamination cycle, (3) a final uranium cycle, dual silica-gel columns, and uranium product storage and shipping facilities, (4) a final plutonium cycle and ion exchange system, (5) a solvent recovery system, and (6) an acid recovery system. At the close of this report period, these modifications were 90% complete.

Equipment modifications in Building 3503 were completed and operations initiated on the Hanford assistance portion of the power reactor fuel reprocessing program. These studies of Redox flowsheets under various conditions were undertaken at ORNL because of the availability of pilot plant equipment and personnel (see "Power Reactor Fuel Reprocessing: Solvent Extraction Studies"). (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Solvent Extraction Studies. — In further tests with soluble poisons for criticality control, boron, cadmium, and rare earths were reduced to 2.5, 17, and 0.2 ppm, respectively, in the uranium solvent extraction product.

The effects of radiation on mixtures of tributyl phosphate, Amsco, and nitric acid are greater than the sum of the effects of radiation on the individual pure components.

Trilaurylamine (TLA), N-Benzylheptadecylamine (NBHA), and dibutyl phenylphosphonate (DBPP) in Amsco were irradiated to 100 to 200 whr/liter. Irradiated 0.1 M solutions of TLA and NBHA, after contact with solutions of uranyl nitrate in nitric acid, clarified nearly as rapidly as unirradiated control samples, but the clarification time of irradiated 0.5 M TLA solutions was greater by a factor of 12. The clarification times of solutions of DBPP in Amsco or Amsco-Tetralin mixtures at concentrations of 15 and 25 g per 100 cc of solution, irradiated to 200 whr/liter, were greater by factors of 2 to 3 than those of unirradiated controls.

Standard Redox flowsheet runs, with both radioactive and nonradioactive feed, were made in Building 3503 to check equipment operability, solution stability, and column flooding limits and to obtain plant scaleup data. A series of Darex and Nyflex runs were made with simulated stainless steel–Redox solutions containing activity obtained from dissolving Graphite Reactor slugs. Throughout these runs, variables such as flow ratios, acid concentrations, and solution component ratios were studied for their effects on losses and decontamination factors. Uranium and plutonium losses ranged from <0.01 to $<0.20\%$ in the extraction column. Gross decontamination factors (based on a feed gamma activity of 10^8 measured by scintillation counter) were 10^3 for the standard Redox flowsheet, 30 to 300 for the Darex flowsheet, and 20 to 60 for the Nyflex flowsheet. In experimental runs in the 2-in. glass columns with Hanford-supplied Nyflex feed, flooding occurred at a flow rate of 660 $gal \cdot ft^{-2} \cdot hr^{-1}$. The aqueous feed solution precipitated in the column, making the column inoperable. (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Sulfex Process. — All major parts of the flowsheet for the decladding of Consolidated Edison fuel with 6 M H_2SO_4 and the subsequent dissolution of the core in 13 M HNO_3–0.04 M NaF–0.04 M $Al(NO_3)_3$ have now been demonstrated on a laboratory scale. The tentative solvent extraction flowsheet for Consolidated Edison fuel will use an acid-deficient first cycle and acid second cycle, with 5% uranium recycle from the second-cycle extraction column aqueous stream back to the first-cycle feed adjustment tank.

Local attack on stabilized Ni-o-nel in Sulfex-Thorex solutions can be largely eliminated by proper heat treatment. During eight Sulfex-Thorex cycles the maximum corrosion rates for this alloy were less than 2 mils/month. Attempts to eliminate the cracking of Carpenter-20Cb without increasing the over-all corrosion rate have not yet succeeded. Pitting attack was observed on Illium 98 in 4 M

and 6 M H_2SO_4. Haynes alloys 25 and 21, high-cobalt alloy types S-590 and and S-816, and Inconel 700 all showed corrosion rates >50 mils/month in boiling 6 M H_2SO_4. (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Zircex Process. — The hydrochlorination of a small piece of an STR subassembly with essentially pure hydrogen chloride (0.02 mole % O_2, 0.01 mole % CO_2) resulted in a uranium loss of 1.92% as HNO_3-insoluble material in the uranium chloride residue. The result of this single test indicates that the use of pure hydrogen chloride alone will not ensure the reduction of insoluble uranium losses to an acceptable value (0.1% or less). (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Zirflex Process. — In several flowsheet demonstration experiments with oxidized unirradiated PWR blanket fuel specimens, the uranium loss to the decladding solution was less than 0.08% and did not vary systematically with the decladding time. The uranium loss to the HNO_3-insoluble residue was less than 0.005% in nearly every case.

In experiments with unirradiated EBWR and STR fuel, as much as 95% and 85%, respectively, of the ammonium fluoride dissolvent have been recycled. The percentages of uranium in the recycle streams were 0.4 and 2%, respectively. (AEC Activity 4301)

Fused Salt—Fluoride Volatility Processing. — The fused salt—volatility process under development for STR fuel consists in dissolution in molten NaF-LiF (42-58 mole %) with anhydrous HF, followed by fluorination below 500°C to remove the uranium with minimum corrosion of the process vessel. In a laboratory hot cell a practice fluorination test at 470°C was successfully carried out; 99.5% of the uranium was volatilized. A test STR fuel dissolution run indicated the formation of zirconium hydride as an intermediate in the hydrofluorination process, as follows:

$$Zr + 4HF \xrightarrow{700°C,\ \text{fused salt}} ZrF_4 + 2H_2$$

$$\tfrac{1}{2}xH_2 + Zr \xrightarrow{\text{HF catalyst}} ZrH_x$$

$$ZrH_x + 4HF \longrightarrow ZrF_4 + (\tfrac{1}{2}x + 2)\,H_2$$

where x is approximately 2. Tests where deliberate hydriding was attempted with HF catalyst tended to confirm this hypothesis. The presence of ZrH_x as an intermediate affects the over-all dissolution rates and possibly interferes with the method of following the dissolution by measurement of hydrogen evolution.

A more detailed study was made of the valence changes in the fluorination of $NaF-ZrF_4-UF_4$ (48-48-4 mole %) at 600°C in a reactor equipped for frequent sampling of the molten salt. At a constant F_2 flow rate of approximately 115 ml/min,

the U^{4+} concentration decreased linearly with time to 0.1 mole %, then approached zero asymptotically. The total U concentration decreased slowly to 3.7 mole %, where the U^{6+}/U^{4+} mole ratio was unity, then dropped sharply to 0.3 mole %, where the U^{6+}/U^{4+} mole ratio was 10/1. Fluorine consumption resulting from vessel corrosion reached a maximum at the U^{6+}/U^{4+} ratio of 1.7/1.

Uranium was successfully recovered from molten $KF-ZrF_4$ (40-60 mole %) by fluorination at 600°C. The initial uranium concentration of 3.7 wt % was reduced to 0.003% in about 15 min. An induction period comparable with that in NaF-bearing salt was observed. This test indicated that the use of KF is possible in fused salt-volatility processing. (AEC Activity 4302)

Waste Treatment. – Continuous laboratory runs demonstrated that 1.36 to 2.40 moles of HNO_3 were destroyed per mole of formaldehyde fed (33 to 60% of the theoretical maximum value). This corresponds to a formaldehyde cost of 6 to 11¢ per gallon for concentrated Purex acid waste, the theoretical minimum being about 4¢. In return for this expenditure the storage volume required for the neutralized waste is reduced about tenfold.

Furnace equipment was assembled for the reduction of wastes to dry solids, and one run was completed in which synthetic neutralized Darex waste was reduced to a fused mass consisting mostly of sodium nitrate.

When Darex waste was calcined at 700°C, the maximum percentages of fission products evolved were 3×10^{-2}% of the beta and 6×10^{-2}% of the gamma. For Sulfex the maxima were 2×10^{-3}% of the beta and 5×10^{-3}% of the gamma. (AEC Activity 4352)

Long-Range Planning Studies. – Samples of U^{233} have been irradiated in the LITR for a period of about seven months, corresponding to 10 to 15% burnup of the original U^{233}. These samples have been removed from the reactor and will be used in a determination of the effective value of α^{23} in an LITR neutron spectrum.

Work has been initiated concerning the value of plutonium as fuel for reactors. The reactors being studied are those presently planned for construction and include APPR, Dresden, Hallam, Yankee, GCR, and Carolina-Virginia reactor types. The value of plutonium will be based on that value which causes plutonium–natural uranium reactors to have the same fuel cost as $U^{235}-U^{238}$ reactors. Reactivity lifetimes and fuel costs are being calculated separately. The reactivity lifetimes have been calculated for Yankee-type reactors. With uranium of 3% enrichment, the lifetime was 5500 Mwd per metric ton of uranium, whereas at 4% enrichment it was 15,600 Mwd/ton. With fuel containing 98% natural uranium and 2% plutonium (the plutonium containing 95% Pu^{239} and 5% Pu^{240}), the lifetime was 5800 Mwd/ton, while with 3% Pu it was 13,500 Mwd/ton. (AEC Activity 4510)

PROGRAM 5000 – PHYSICAL RESEARCH

Reactor Operations. – In the current three-week operating cycle of the ORR, the number of scrams was reduced to approximately 4 from an average of about 12 in previous cycles; in no case was startup of the reactor prevented by the buildup of xenon following a scram. One scheduled reloading was necessary since the reactivity decreased below the limit required for operation. It appears that a mid-cycle reloading will be necessary for 20-Mw operation.

A number of flux-trap experiments have been done in the ORR. In these experiments, the reactor lattice was arranged to give a beryllium core surrounded by a single row of fuel, with a row of beryllium outside the fuel. More of these experiments will be done in December to complete the series, but it now appears possible to reach, or perhaps exceed, 10^{15} neutrons$\cdot$cm$^{-2}\cdot$sec^{-1} in the center of the core at 20 Mw.

The air coolers at the ORR, previously reported as deficient in cooling capacity, have been damaged in several cases by vibration of the turbulators inside the tubes. This has worn holes in the tube walls in at least two cases. Negotiations are under way with the manufacturer to correct this situation, and the turbulators have been removed completely from six of the eight units.

Engineering is under way on two methods of annealing the stored energy in the Graphite Reactor. This will permit the two methods to be compared on the basis of down time, costs, and safety. Reloading of the Graphite Reactor is delayed pending a review of the effects on the experimental program, particularly the work in the Solid State Division.

PHYSICS

Neutron Velocity Selector. – The neutron transmission of a sample of Pu^{240} (enriched to 92.19%) was measured in the thermal energy range. A preliminary value of 285 ± 8 barns for the 2200-m/sec absorption cross section was obtained after the data were corrected for the contributions of the other plutonium isotopes and an assumed 10-barn scattering cross section. The cross sections of enriched samples of the odd-odd nuclides Lu^{176} (enriched to 70.2%) and Ta^{180} (enriched to 0.25%) were measured up to 43 and 11 ev, respectively. Twelve levels have been assigned to Lu^{176} at 0.142, 1.57, 4.35, 6.13, 9.77, 10.8, 11.5, 11.9, 19.1, 19.9, 21.8, and 24.6 ev, with six more resonances observed up to 43 ev. In addition to the resonance at 0.43 ev previously assigned to Ta^{180}, two new resonances at 2.13 and 5.96 ev were observed. A sample of Lu^{175} was also prepared and measured to correct for the Lu^{175} resonances which were observed in the Lu^{176} sample. Thirteen resonances have been assigned to Lu^{175} up to 43 ev. From these data, level spacings per spin state of 4.8 ± 0.8 ev for Lu^{176} and 7.1 ± 1.5 ev for Lu^{175} have been computed. Samples of Np^{237}, Am^{241}, and Tc^{99} were measured up to 36, 10.3, and 454 ev, respectively. Thirty-two levels were observed in

Np^{237}, leading to a strength function $(\overline{\Gamma_n^o}/D)$ per spin state of $(1.1 \pm 0.2) \times 10^{-4}$ up to 24 ev and an average level spacing per spin state of 1.5 ± 0.3 ev up to 12 ev. Ten definite and three probable resonances were observed in the Am^{241} measurements, leading to a strength function of $(1.7 \pm 0.5) \times 10^{-4}$ up to 10.3 ev and an average level spacing per spin state of 1.4 ± 0.4 ev up to ~7 ev. The Tc^{99} sample exhibited 14 resonances and has a strength function of $(0.45 \pm 0.17) \times 10^{-4}$ up to 300 ev and an average level spacing of 40 ± 10 ev per spin state up to 200 ev. (AEC Activity 5220)

High-Voltage Experimental Program: Measurement of the Longitudinal Polarization of Beta Particles from P^{32} Decay. — Longitudinal spins of beta particles are precessed by deflection through 136° in a spherical electrostatic spin rotator. The transverse component of polarization is analyzed by 135° Mott scattering from thin gold foils. Scattered electrons are detected with two multichannel anthracene scintillation spectrometers. Tests with the 624-kev K-conversion line of Cs^{137} showed that this rotator has an energy resolution of 4% and a transmission of $\sim 2 \times 10^{-4}$ for a ±6° azimuthal acceptance angle at the source. Measurements were made on P^{32} sources having strengths of 60 and 1200 mc (0.3 and 4 mg/cm^2, respectively, and 2 mm in diameter) at 624 kev (electron velocity 0.89c). For a 1.00-mg/cm^2 gold scattering foil, the weak and strong sources gave the same asymmetry to within the standard error of the comparison, which was ±1%. The strong source was also used with six other gold foils ranging from 0.1 to 1.5 mg/cm^2. The seven values of asymmetry, when plotted against foil thickness, fall on a straight line that extrapolates to 0.484 ± 0.003 at zero foil thickness. This corresponds to a polarization of -0.99 v/c. The standard error, including some of the systematic errors, is ±2%. Other possible sources of error are being studied. (AEC Activity 5220)

High-Voltage Experimental Program: Neutron Absorption Cross Sections at 27 kev. — The neutron absorption cross section has been measured at 27 kev for several elements by a spherical shell transmission method. Neutrons were obtained from a source consisting of a solid antimony sphere encased in a thin (0.030-in.) spherical beryllium shell. The average neutron energy from this source was 27 kev; the spread (full width at half maximum) was 3 kev. The neutron detector was a modified long counter, shielded from source gamma rays by lead; this entire assembly was positioned at a distance from the source that was large as compared with the sample shell diameters. A serious attempt was made to maintain good geometry in these measurements, and corrections for finite source size, finite source-to-detector distance, etc., were minimized, evaluated, and taken into account. Multiple scattering in the sample shells was evaluated according to the method of Bethe, Beyster, and Carter (LA-1429). Preliminary absorption cross sections for elements measured thus far are as follows: silver, 940 ± 70 mb; gold, 520 ± 100 mb; antimony, 500 ± 100 mb; lead, 10 ± 20 mb. (AEC Activity 5220)

High-Voltage Experimental Program: Large Liquid Scintillator for Neutron Capture Studies (29 kev). — A 1-m-dia liquid scintillator tank was constructed and used with the ORNL 3-Mv pulsed Van de Graaff accelerator. Over-all time resolution is 15 to 20 mμsec. Energy resolution on a single gamma-ray peak is

25 to 30%. By use of "kinematically collimated" neutrons from the $Li^7(p,n)$ reaction (peak intensity at 29 kev; full width at half maximum, 17 kev) the radiative capture cross sections for a number of elements have been compared. Absolute cross sections have been assigned by normalization of the absorption cross section of silver as measured by shell transmission at 27 kev (Sb-Be neutrons). Pulse-height spectra in the scintillator have been studied for neutron capture in 19 of the elements studied. Conversion of the full binding energy to a light pulse, even with over 40 cm of gamma absorption path in the scintillator, seems to be relatively rare. The spectra are sufficiently characteristic, however, to identify neutron binding energies. Values of 6.5 ± 0.4 Mev for Tb^{159} and 6.2 ± 0.4 Mev for Ho^{165} were found in this way. (AEC Activity 5220)

High-Voltage Experimental Program: Neutron Capture Cross-Section Calibration at 63 kev. — The flat-response 4π graphite-sphere neutron detector (Nuclear Instr. 1, 335–339, 1957) has been used to establish the absolute neutron flux used in the capture-cross-section experiments at 29 kev. This was accomplished by measuring the 54-min beta activity induced in indium samples. A ZrT target was also used to provide a collimated beam of neutrons from the T(p,n) reaction, of average energy 63 kev (full width at half maximum, 40 kev). With the assumption that the response of the neutron detector is uniform, the 29-kev absolute cross sections were extended to this energy. Comparison of the direct capture gamma cross sections with shell transmission and with activation results shows agreement in many instances but disagreement in a few cases. (AEC Activity 5220)

Charge Spectrometry. — The momentum spectrum of singly charged Li^6 recoils following the beta decay of He^6 is being studied with the spectrometer which was used for an earlier investigation (Phys. Rev. 100, 1396, 1955) of A^{37}. The He^6 is generated in BeO powder by the $Be^9(n,\alpha)He^6$ reaction at the ORR. Water vapor carries the He^6 to the spectrometer, where the He^6 is left to decay in a field-free conical source volume. The spectrometer has magnetic and electrostatic deflectors, in series, which eliminate any simultaneous observation of singly and doubly charged Li^6 ions. Three stages of differential pumping make it possible to count with a signal-to-background ratio of 4 at the peak of the charge-1 spectrum. The upper 60% of this momentum spectrum has been observed and agrees well in shape with the theoretical curve for a pure axial-vector interaction. The charge-2 spectrum exhibits the same shape within the statistical uncertainties (±5%). Comparison of intensities at 1000 ev yields the value (9.5 ± 0.5)% for the abundance ratio of charge 2 to charge 1 plus charge 2; the theoretical value of this ratio is (10.5 ± 1.5)% (Kgl. Danske Videnskab. Selskab, Mat.-fys. Medd. 27, No. 2, 1952). The observed end point of the recoil spectrum corresponds to a beta-decay energy of 3.52 ± 0.02 Mev. (AEC Activity 5220)

Neutron Diffraction. — Neutron diffraction investigations were performed on metallic cerium in an attempt to clarify the anomalous behavior which was observed in previous specific heat and magnetic susceptibility measurements. Most of the difficulty in the interpretation of these macroscopic results has arisen because the exact crystal structure of the samples has not been understood. There are three

crystallographic forms of cerium that exist between 450 and 4°K, and the relative concentrations of the three phases are dependent both on the sample temperature and on the previous history of the sample. The neutron diffraction results have made possible a correlation of the interesting magnetic behavior with the existing crystallographic conditions. Experiments at various temperatures from 300 to 4°K on three specially prepared specimens indicate that there is a change in the electronic configuration of the cerium atoms when the collapsed face-centered cubic phase is formed and that there is an antiferromagnetic transition in the hexagonal close-packed phase at a temperature near 13°K. (AEC Activity 5220)

Low-Temperature, Nuclear and Solid State Physics. — Recent measurements involving the use of helium as a counting gas have shown that W (average energy to form an ion pair) is very sensitive to traces of foreign gases. For the investigation reported here, a small gridded parallel-plate ionization chamber was operated in helium vapor at 2 to 2.6°K, where all materials except helium have vapor pressures less than 10^{-12} mm Hg. The feasibility of helium vapor pulse chambers has previously been demonstrated at ORNL (Rev. Sci. Instr. 26, 521, 1955); however, no detailed measurements were reported. Alpha particles from Am^{241} were collimated with paths approximately parallel to the electrodes. The data were normalized by using a counting gas consisting of 90% argon and 10% methane at 300°K. The rise time, corrected for amplifier clipping, was ~10 to 20 μsec with a grid-plate spacing of 0.95 cm and appeared to vary slowly when the electrode potentials were varied by a factor of 30. If the pulse-height data are extrapolated to zero gas density and W_{A-CH_4} is taken to be 26 ev, the value of W_{He} is found to be $\leq 46 \pm 4$ ev. This agrees well with the value of W_{He} given by others (Phys. Rev. 90, 1120, 1953; Phys. Rev. 93, 1236, 1954). (AEC Activity 5220)

CHEMISTRY

Druhm Process. — Three experimental reduction runs were made with three different types of UF_6 nozzles. A helium-cooled copper nozzle showed favorable corrosion resistance to the reactor environment. Of the uranium introduced to the reactor as UF_6 in run D-17, 88.3% was recovered as consolidated uranium metal having a maximum purity of 99.6%. (AEC Activity 5310)

Raw Materials Research and Development: Amine Extraction. — In the extraction of neptunium(IV) from nitric acid solutions by triisooctylamine in xylene, the extraction coefficient increased rapidly with nitric acid concentration up to 1 M and passed through a maximum at ~2 M. From nitrate-nitric acid solutions, the extraction coefficient at 1 M HNO_3 was proportional to the square of the nitrate ion concentration; at 6 M NO_3^-, the coefficient was inversely proportional to the 3/2 power of the nitric acid concentration over the range 1 to 6 M. Extractions with secondary amines were lower, and with quaternary ammonium salts they were higher at nitric acid concentrations of 2 M and greater. For extraction by di(2-ethylhexyl)phosphoric acid, the coefficient was inversely proportional to the square of the nitric acid concentration (approximately) over the range 1 to 10 M;

below 1 M, varying hydrolytic effects sometimes increased and sometimes decreased the extraction. From solutions 6 M in nitrate ion, the extraction coefficient increased slightly with the nitric acid concentration up to 0.5 M, then decreased, varying inversely as the square of the nitric acid concentration over the range 0.5 to 6 M.

For the simultaneous extraction of neptunium(IV) and plutonium(IV), the neptunium(V) and (VI) can be quantitatively reduced to neptunium(IV) by ferrous sulfamate at nitrate concentrations of 5 M or higher. Plutonium(III) can then be oxidized to (IV) by nitrite without reoxidation of the neptunium.

In continued study of structural effects in other trialkyl phosphates considered as alternates for tributyl phosphate (TBP) (ORNL-2595), separation factors for uranium from thorium increased slightly with increased branching of primary alkyl radicals. The separation factors increased by about a factor of 10 over those obtained with TBP when the primary alkyl radicals were replaced by sec-butyl and sec-capryl. Separation factors from fission products (gross beta and gamma) in batch test were improved by factors of 5 to 10. These secondary alkyl phosphates share the advantage shown by some other branched-alkyl phosphates (ORNL-2595) – that the dialkylphosphoric acids which are the expected main degradation products do not precipitate thorium. (AEC Activity 5310)

Raw Materials Research and Development: Dapex Process. – Recovery of uranium from bomb-liner salvage was demonstrated with the Dapex process in a continuous run. The uranium was extracted from a sulfuric acid leach slurry (20 wt % solids) in five stages; the recovery of uranium was 99.8%. The uranium was stripped from the solvent with ammonium carbonate and then precipitated with ammonia. The product was free of sodium and low in sulfate. Virtually no emulsions were encountered, and the solvent loss to the slurry raffinate was <0.6 gal per 1000 gal. (AEC Activity 5310)

Chemical Engineering Research. – A diffusion cell system is being developed to determine experimentally the interface resistance to diffusion in the extraction of uranyl nitrate into tributyl phosphate. Calculations showed that interface reactions having fictitious homogeneous half times of 1 msec will present resistances equivalent to the diffusion of solute through about 0.1 mm of solvent and can be easily detected in the cell. (AEC Activity 5310)

Equipment Decontamination. – The Turco 4501 process (developed by Turco Products, Inc.) was used for the removal of Nb^{95} from types 304 and 347 stainless steel coupons; the treatment gave decontamination factors of 11 to 680, leaving less than 0.5 mr/hr on the metal. Controls in which similar coupons were contacted with boiling 8 M HNO_3 for 1 hr gave decontamination factors of 1.0 to 2.8. Treatment of a short section of stainless steel pipe from the HRT by the Turco 4501 process removed the corrosion scale and reduced the gamma contamination from 5.4 r/hr to 95 mr/hr. Repeating the treatment further reduced the activity to 3 mr/hr. The residual activity consisted of Ru, Nb, Fe^{59}, and possibly Cr^{51}.

Alkaline periodate solution reduced Nb^{95} contamination on type 347 stainless steel from 14 mr/hr to 0.6 mr/hr. However, this solution was not effective for descaling or decontaminating a section of HRT pipe. (AEC Activity 5310)

Transuranics Recovery. — An extraction process which may be useful for recovering plutonium and transplutonic elements from irradiated plutonium was tested in a laboratory batch countercurrent extraction with synthetic feed solution containing tracer plutonium, americium, and yttrium. The aqueous feed solution, 3 M in NH_4OH, 1 M in $Al(NO_3)_3$ and 0.1 M in $NH_2OH \cdot H_2SO_4$, at pH 1.5, was contacted with 40% tributyl phosphate (TBP), and the plutonium(III), americium, and yttrium tracers were extracted. With six stages, the losses were about 0.01%. Contacting the organic phase with 6 M HNO_3 containing 0.1 M $NaNO_2$ oxidized the plutonium to the tetravalent state, so that it was retained in the organic phase, while the trivalent americium and yttrium were transferred to the aqueous phase. With three partition stages and two organic scrub stages, 98.6% of the americium and yttrium and only 0.001% of the plutonium were transferred to the aqueous phase. The plutonium stream and the transplutonic-rare-earth stream could then be treated separately for further purification.

Scouting tests show that americium-yttrium separation factors of about 8 can be obtained in a single stage when 1 M NH_4CNS is contacted with 2 to 35% TBP. Separation factors of about 4 were obtained when 0.1 to 1 M HNO_3 was contacted with 0.5 M trioctylphosphine oxide in xylene. (AEC Activity 5310)

Feed Materials Processing. — Uranium metal produced by the reduction of UF_6 by lithium amalgam contained less oxygen, hydrogen, nitrogen, and iron than electrolytic-grade metal. Contaminants due to the processing method, lithium and fluoride, were <20 ppm. The mercury content was 280 ppm, but this was thought to be due to recontamination of the small uranium sample from the atmosphere of the vacuum distillation retort on cooling. (AEC Activity 5310)

Chemistry of Corrosion. — In the study of the mechanism of inhibition it was shown that an aerated solution containing 0.01 f benzoic acid neutralized to a pH of 5.55 passivates iron to potentials above the Flade potential. As was found with the XO_4^{n-} inhibitors, the passivation is sensitive to addition of sulfate ions, and the falling potential halts in the region of the calculated Flade potential before passing to values in the active region. This is convincing evidence that inhibition does not necessarily require that the inhibitor itself possess precipitating or oxidizing properties with respect to ferrous ions, but, rather, that it may also function by changing the kinetics of other possible oxidation-reduction processes in the system.

The study of the formation of thin films on zirconium was continued with potentiostatic measurements in an aqueous environment free from oxygen or any other effective oxidizing agent. Under these conditions, the rate of film formation is given directly by the current passed through the cell, and the rate-time curve may therefore be determined with considerably more precision than with the indirect polarization method used previously. The rate-time expressions obtained were

essentially identical with those obtained by the polarization method in an oxygenated environment. These results support the validity of the polarization method of measuring rates and confirm the kinetic analysis of film formation previously reported. (AEC Activity 5330)

Characterization of Short-Lived Fission Products. — The yield of 7.8-min Ga^{74} in the fission of U^{235} in the Graphite Reactor was determined as 3×10^{-4}%. This estimate was obtained by comparisons of the intensities of the prominent 0.60-Mev gamma ray of Ga^{74} and the 0.54-Mev gamma ray of 12.8-day Ba^{140}; both the gallium and the barium were isolated from the same irradiated uranium. The absolute intensities of the 0.60- and the 0.54-Mev transitions were taken as 100 and 21.5%, respectively. The above yield value for mass number 74 is in good agreement with an extrapolation of the smooth mass–fission-yield curve for U^{235}. (AEC Activity 5330)

High-Temperature Aqueous Solution Chemistry. — The comparative osmotic behavior of duplicate samples of the following salts was investigated by means of isopiestic vapor pressure measurements as a function of concentration at 99.60°C: LiCl, NaCl, KCl, CsCl, $BaCl_2$, $MgSO_4$, Na_2SO_4, UO_2SO_4.

These salts were compared with KCl solutions that varied in concentration from 0.5 m to more than 4 m. Concentration intervals of 0.5 m were employed. Duplicate samples agreed in the poorest cases within 0.4% in the final calculated molality. This latter value includes the total integrated error from all factors affecting the osmotic method at elevated temperatures. In many cases, duplicate samples agreed within 0.1%. (AEC Activity 5330)

Nuclear Methods of Chemical Analysis. — A preliminary investigation has been made into the feasibility of using short-half-life radionuclides in activation analysis. A typical analysis involves the irradiation of a specimen in the pneumatic tube of the ORNL Graphite Reactor for 1 min, after which the radioactive material is analyzed by means of gamma spectrometry. The results obtained so far in this investigation are summarized below. The elements listed were determined in samples of metals, alloys, biological specimens, and similar materials.

Element	Radionuclide	Half Life (min)	Gamma Radiation Energy (Mev)	Limit of Measurement* (μg)
Co	Co^{60m}	10.5	0.059	2.0
Rh	Rh^{104m}	4.4	0.053	0.1
Pd	Pd^{109}	4.8	0.19	10.0
Pt	Pt^{199}	31.0	0.070	100.0
Cu	Cu^{66}	5.1	1.04	5.0
Sn	Sn^{125}	9.5	0.15, 0.32	10.0
Ti	Ti^{51}	5.8	0.32	2.0
Al	Al^{28}	2.3	1.78	1.0

*Based on conditions involving neutron flux, time from reactor discharge, and counting efficiency. (AEC Activity 5330)

Chemical Separation of Isotopes. — Equilibrium constants have been measured as a function of temperature for the fractionation of boron isotopes between BF_3 gas and the 1:1 addition compounds of BF_3 with di-n-butyl sulfide and diethyl sulfide:

$$B^{10}F_3(g) + B^{11}F_3 \cdot RS(l) \rightleftharpoons B^{11}F_3(g) + B^{10}F_3 \cdot RS(l)$$

The results are tabulated below:

Butyl Sulfide		Ethyl Sulfide	
Temperature (°C)	K	Temperature (°C)	K
-20	1.054	-20	1.059
- 3	1.044	-10	1.053
12	1.032	0	1.050
25	1.033	10	1.048

(AEC Activity 5340)

METALLURGY

High-Temperature Reactions of Metals and Ceramics. — The reciprocal salt system consisting of the chlorides and nitrates of lithium and potassium was shown to be volumetrically ideal for all binary compositions and also for the four ternary compositions for which measurements were made. By volumetric ideality it is meant that the partial molal volumes $\bar{V}$ are constants independent of concentration and equal to the equivalent volumes of the pure constituents. It was found that the partial molal volumes of lithium chloride and potassium chloride for compositions at which these pure constituents are solids can be accurately computed by assuming the density functions to be valid in the supercooled region.

It was shown that this system exhibits a higher order of volumetric ideality in that there exists an additivity relation among the four partial molal volumes as functions of temperature such that any three of these quantities specify the fourth. (AEC Activity 5420)

Radiation Effects on Oxidation. — A study is in progress to determine the influence of radiation on the degree of protectiveness of oxide films on niobium. It was previously shown that at 400°C niobium forms an initially protective oxide film which becomes nonprotective with increasing time because of the formation of microscopic cracks in the oxide. A series of niobium specimens has been oxidized in the neighborhood of 400°C in the ORNL Graphite Reactor in a neutron flux of about 6×10^{11} neutrons $\cdot$ $cm^{-2} \cdot sec^{-1}$. The results were very similar to those obtained with unirradiated specimens. Crack formation occurred in the oxide on the irradiated specimens in about the same time as for the unirradiated samples,

and the surface topography of the oxide prior to cracking was identical on the two sets of specimens. It has not yet been determined, however, whether or not a change in the rate of oxidation of the irradiated samples occurred before crack formation was initiated. (AEC Activity 5420)

PROGRAM 6000 – BIOLOGY AND MEDICINE

BIOLOGY

Cytology and Genetics: Paramecium. – Earlier work on the postirradiation development of mutation in Paramecium has led to the hypothesis that there is a progressive loss of initial radiation damage until some critical moment at which the remaining damage is converted into final mutation. The evidence has suggested that the critical moment is some event in the normal development of well-fed paramecia for division. As was reported earlier, when starved paramecia are irradiated, less mutation is detected the longer the interval between irradiation and addition of food. This loss was shown to continue for at least six and probably more hours. It has now been shown to continue beyond 24 hr, and there is no reason to think that it does not continue indefinitely until all the initial reversible or reparable damage is lost. This confirms and strengthens the earlier conclusion that the conversion of initial damage to final mutation is brought about by some event in the development of the paramecia for division, since as long as the paramecia remain in the stationary phase the conversion apparently does not take place. There is some indication, however, that some part of the initial damage is not reparable or reversible and that 48 hr of starvation after irradiation allows the majority of the reparable or reversible kind to be lost. (AEC Activity 6130)

Cytology and Genetics: Neurospora and Schizophyllum. – Complementation of mutants in the ad-3 region of Neurospora crassa is being studied further by combining some 200 purple adenine mutants in all possible combinations in dikaryon. About 50% of the 40,000 possible combinations have been made to date. Whereas such tests previous to the present analysis indicated that only ad-3A and ad-3B mutants could complement in a dikaryon, the present analysis shows that complementation is not restricted to dikaryons of this type. A certain class of ad-3B mutants when combined with another class of ad-3B mutants can also complement to give dikaryons which grow at wild-type rate.

The effects of diepoxybutane and ultraviolet light are being studied in reverse mutation experiments with Neurospora. It was earlier observed that diepoxybutane followed by ultraviolet greatly enhanced the frequency of reversions induced in the adenineless mutant 38701. This enhancement was expressed as a mutation frequency higher than that which would be expected if the two treatments were additive. In order to get a clearer picture as to how much of the combined effect was due to each of the single treatments, experiments are now being carried out where conidia given the single treatments are kept in water at different temperatures and for

varying lengths of time before they are plated. From this it was found that there is a long-lasting increase in mutation rates after the diepoxybutane has been washed out. This effect is continued for several hours without any decrease in survival. A similar effect could not be detected after treatment with ultraviolet light. (AEC Activity 6130)

Cytology and Genetics: Bacteriophage. — Research summarized earlier (ORNL-2593, p 29) has shown that bacteriophage T4r mutants can be induced to revert to T4 wild type as a result of ultraviolet irradiation and multiplicity reactivation of extracellular phage. Two questions bearing on the interpretation of that observation are currently under investigation: (1) Is the phenomenon unidirectional, or can a similar treatment of T4 wild-type phage induce mutation to T4r; and (2) is multiplicity reactivation of killed phage essential to the process of ultraviolet-induced genetic change? Preliminary experiments now indicate that mutation can be induced in either direction and that fewer, if any, induced mutants are found among the progeny of phage surviving a low dose of ultraviolet than in the progeny produced by multiplicity reactivation of the same irradiated phage population. (AEC Activity 6130)

Cytology and Genetics: Insects. — The primary radiation lesion in gametes of the wasp Habrobracon results in death during early embryogeny. The syndrome is a specific one — nuclei are produced that are Feulgen negative, and these eventually swell up to 200 times the normal size. It has been impossible to mimic this lesion by genetically contriving the loss of chromosomes or chromosome parts. However, a mutation has been recently discovered that mimics the primary radiation lesion when the female is homozygous for the mutation. Close study of the wasp during the egg-laying process revealed that the eggs did not pass down the ovipositor, a condition thought to be necessary for normal development since the egg is distorted in such a way that the egg nucleus passes from the egg interior to the periphery, where normal development begins. Indeed, it was found that in eggs from the mutant wasp the nucleus remained in the egg interior and development did not begin. After a few hours an abortive division took place, and then Feulgen-negative nuclei were produced that eventually swelled.

It now appears likely that radiation acts by blocking normal nuclear division even though the egg-laying process is normal. Eventual imbalance from differential embryogenesis of the cytoplasm and nucleus causes the observed syndrome. (AEC Activity 6130)

Pathology and Physiology. — Immunogenetic studies of mouse hemoglobin are being made with reference to its possible use as a marker in the transplantation of bone marrow. Gluecksohn-Waelsch et al. have recently described inheritable hemoglobin differences in mice (J. Clin. Invest. 36, 753–56, 1957). The mouse hemoglobins examined by them with paper electrophoretic techniques were either single and homogeneous (designated type I) or diffuse and apparently heterogeneous (type II). Genetic analyses suggested that types I and II segregated as simple Mendelian autosomal alleles, type II being dominant. After starch-gel electrophoresis (Biochem. J. 61, 629–41, 1955), a single band is found for type I hemoglobin, but type II

hemoglobin resolves into three electrophoretically separable fractions. In starch gels, the electrophoretic mobility of type I differs from the electrophoretic mobility of all three fractions of type II; however, the absorption spectra (9000–2200 Å) of the different hemoglobins appear to be similar. Other physicochemical properties are now being studied. Genetic linkage tests show that the hemoglobin locus is in the first linkage group and is fairly tightly linked with albinism. Furthermore, data obtained from mice segregating from these traits indicate that the positions of the loci are related to one another in the following order: pink eye, chinchilla, and hemoglobin type. "Recombinant" hemoglobin patterns were not observed in any of approximately 500 F_1 intercross and backcross progeny examined; thus it is not yet known whether the hemoglobin locus is simple or compound. (AEC Activity 6130)

Biophysics: Electron Spin Resonance Studies. — Investigation of the lifetimes, the dependence of the fading or decay of the radiation-induced magnetic centers on temperature, and radical induction as a function of linear energy transfer (LET) have been continued. The variation of induced resonances in crystalline amino acids with LET follows closely the induction of color in fluorescence centers in irradiated glasses. Since all the present results are compatible with both a lattice defect theory of the origin of the magnetic centers and a simple free-radical hypothesis, attempts are now being made to determine which of these alternative mechanisms is of greater importance in irradiated solids. The studies now in progress involve the irradiation of amino acids dispersed in gels and the investigation of magnetic resonances induced under such conditions. (AEC Activity 6130)

Biophysics: Silver-Activated Phosphate Glass. — The readings of phosphate glass rods exposed to Po^{210} alpha particles, 14.1-Mev neutrons, and P^{32} beta rays were all found to increase linearly with increasing doses. This result indicates the versatility which may be expected from this small glass dosimeter. The fluorescence output of the glass per unit of absorbed energy was compared among Po^{210} alpha particles, 14.1-Mev neutrons, recoil protons produced by 14.1-Mev neutrons, P^{32} beta rays, and Co^{60} gamma rays; the output was found to decrease with increasing linear energy transfer of the radiation. Rods implanted in a mouse showed that the internal dose distribution inside the mouse is homogeneous within 10%. The improvement of the accuracy is under way. (AEC Activity 6130)

Biophysics: Photoprotection Studies with Bacteria. — Photoprotection (PP) is a phenomenon in which damage by ultraviolet light (UV) is decreased by administering visible light before UV irradiation. Studies conducted with *Escherichia coli* B have shown that PP occurs both when the bacteria are plated on plain nutrient agar and when they are plated on agar containing NaCl; however, the effect is much greater in the latter case. At high doses of UV (10^{-3} survival on salt agar), PP disappears. If cells are held in phosphate buffer at 23°C, PP reaches a maximum 30 min after illumination, but some protection is retained for as long as 4 hr. In these experiments, PP is complicated both by dark protection and by lethal effects of the illumination. (AEC Activity 6130)

Microbial Protection and Recovery: Radiation Studies on a Porphyrin-Requiring Strain of Escherichia coli. — Efforts to understand the mechanism by which the sensitivity to hydrogen peroxide is increased in irradiated cells of this organism are being continued. Irradiated extracts of these catalase-negative cells have proved to be very efficient in bringing about cell inactivation. This suggest a role for organic peroxides, possibly produced in equilibrium with H_2O_2.

It was established that these catalase-negative cells have no flavoprotein system for decomposing H_2O_2. This result is important because it removes one of the possible explanations for sensitization. Current experiments indicate that the medium in which the cells are grown prior to irradiation has a dramatic effect on the development of H_2O_2 sensitivity. It is possible to prevent the development of sensitivity by growing the cells in a high-glucose medium. (AEC Activity 6230)

Microbial Protection and Recovery: Biochemical Studies on the Porphyrin-Requiring Escherichia coli. — Growth experiments with E. coli H7 have shown that the organism has a greatly increased lag phase when cultured in a chemically defined medium in comparison with growth in a peptone-glucose medium. The growth requirements in the defined medium were investigated. When the organism was cultured aerobically in the absence of hemin, an acetate and cysteine requirement was observed. The sulfhydril amino acid was found to supply the oxidized disulfide form, cystine, and in the presence of the latter there was no stimulation by cysteine. The possibility that cystine diminishes the lag phase by reducing the oxidation-reduction potential of the medium is being investigated. The chemically defined medium as it is presently constituted permits growth that is comparable in rate and final titer with growth in the complex medium.

The growth rate in the peptone medium when 0.5% glucose was supplied was the same anaerobically in the absence or presence of hemin, as well as aerobically when hemin was supplied. The aerobic cultures lacking hemin have a slower growth rate and lower final titer. A 20-liter culture was grown anaerobically in the presence of hemin, and the cells harvested from this culture were of two kinds, small brown and normal-size white. These cells were easily separated by centrifugation, and both were found to be catalase positive. Manometric studies of the cells grown in the presence and absence of hemin showed marked differences in their metabolism. (AEC Activity 6230)

Mammalian Recovery. — Histological and cytological changes in lymphatic tissues are being related to the foreign bone marrow reaction that causes the death of many irradiated mice treated with foreign marrow.

Repopulation of the bone marrow of lethally irradiated mice by homologous marrow is accompanied by a more severe anemia than that seen in control animals treated with isologous marrow.

Following a lethal dose of x rays, there is a loss of intestinal weight. The radiation-protective compound S,2-aminoethylisothiourea (AET) however, was found to largely offset this loss in the irradiated mouse. The protection of other gastrointestinal organs by AET is also being studied.

Techniques for preparing cellular suspensions of various mouse tissues were developed. Transplantation of these tissues, namely, testis, intestinal epithelium, liver, and pancreas, by the intravenous route is being tested.

The mechanism of the regeneration of intestinal epithelium after irradiation of the mouse has been investigated. The results indicate that the epithelial cells differentiate abnormally (hypertrophy), instead of flattening out as previously proposed by other workers. (AEC Activity 6230)

Radiation Immunology. — Previous studies on the development of the antibody-forming mechanism in mice showed a subnormal immune mechanism in mice from two to six weeks of age when compared with 12- and 30-week-old adult mice. Further studies have shown that mice nine weeks of age are also immunologically immature. The development of a recognition mechanism in the young mice was made apparent when sheep, rat, and rabbit red blood cells were used as antigens; mice from two to six weeks of age showed a differential response in favor of the more distantly related antigens (sheep > rabbit > rat), whereas adult mice respond equally to these three antigens. Preliminary studies also indicate that the antibody production in young mice is dependent upon the dose as well as the nature of the antigen used for immunization.

Studies of the relation of secondary antigen injection to time of irradiation on antibody production in mice have shown that a maximum depression in the secondary response occurs when antigen is injected one day before to 1 hr after x irradiation, and a significant depression is not apparent when antigen is injected ten, five, or even three days before exposure. Also, recovery appears to take place in ten days. In contrast, previous studies on the primary antibody response showed that a maximum depression occurs one day after x irradiation and that recovery takes place eight or more weeks after exposure. (AEC Activity 6230)

PROGRAM 8000 — WORK FOR OTHERS

Reactor Operations Supervision. — The committee on admissions reviewed applications and determined that 21 applicants from 12 foreign nations were qualified. Final selections to fill the ten openings will be made by the Division of International Affairs, AEC. (AEC Activity 871x)

Reactor Hazards Evaluation. — The committee on admissions reviewed applications and determined that 16 applicants from 11 foreign nations were qualified. The maximum permissible enrollment in this course is 16. (AEC Activity 871x)

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