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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

MARCH 1960

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

MARCH 1960

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Oak Ridge, Tennessee
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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

March 1960

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

PROGRAM 2000 – SPECIAL NUCLEAR MATERIALS

Power Reactor Fuel Processing Pilot Plant. – Decontamination of Building 3019 was continued. The attic and the sampling gallery were decontaminated to an average transferable level of < 30 d/min per 100 cm^2 and to an average direct-reading level of < 300 d/min per 100 cm^2. Three coats of "color coded" paint were applied to immobilize the residual plutonium activity. The new stainless steel floor in the operating gallery was completed. The ceiling and the walls of the operating area above the cells (the penthouse) were decontaminated to an average transferable level of 45 d/min per 100 cm^2 and an average direct reading of 1800 d/min per 100 cm^2; decontamination is continuing in order to reduce the remaining spots of activity to < 3000 d/min per 100 cm^2. (AEC Activity 2461)

PROGRAM 4000 – REACTOR DEVELOPMENT

GAS-COOLED REACTOR PROGRAM

Thermodynamics. – Calculations have been completed for determining the expected EGCR control rod surface temperatures. The current core design provides for a flow of 100 lb/hr up through the center of the control rod and 25 to 75 lb/hr flow in the annulus between the rod and the walls of the hole, depending on the rod position in the core. Heat transfer calculations have been made for the case of a control rod fully inserted in the center of the core, in which the average heat generation rate is 8570 $Btu \cdot hr^{-1} \cdot ft^{-1}$, with a peak generation of 12,800 $Btu \cdot hr^{-1} \cdot ft^{-1}$. Allowances were made for heat transfer by radiation to the graphite and for the changes with temperature level in the clearances between the B_4C and the cladding. Heat generation rates over the length of the rod were based on the axial flux gradient in a core with the inverted fuel loading scheme and with control rods inserted 9 in. The results of the calculations indicate that for 100 lb/hr flow the cladding temperatures will be below the allowable limit of 1600°F. Also, since no credit was taken for convective cooling in the annulus, the cladding temperatures are somewhat high. An increase in the emissivity of the outside stainless steel cladding from 0.3 to 0.6 decreases

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the maximum cladding temperature by about 150°F. The average amount of heat transferred by radiation is 45% for the case of an emissivity of 0.6 and 37% for the lower value of 0.3. (AEC Activity 4141.1)

GCR-ORR Loop No. 1. – Pressure vessel tests were completed on the heater and cooler components of the loop. These units were tested at their rated temperatures and pressures plus 100 psi for 30 min. The in-pile section, including the transfer station, was tested at 600 psig (1.5 times design pressure) at room temperature for 30 min. All test results were satisfactory. Preliminary analyses of results of the completed thermocouple calibration tests indicate that fuel element cladding temperatures can be predicted within reasonable limits from the gas stream temperatures. (AEC Activity 4141.1)

Irradiation Experiments. – Fission gas release data for several capsules irradiated in the LITR are given below:

Capsule No.	Maximum UO_2 Temperature (°F)	Burnup (Mwd/metric ton)	$\int k\, d\theta$ (w/cm)	Fission Gas Released (%)	
				Xe^{133}	Xe^{135}
L-10a	2450	1600	1.8	Ruptured in reactor	
L-10b	2900	1600	1.8	29	26*
L-16a	1950	2900	1.0	0.078	0.021
L-16b	2050	2900	1.0	0.033	0.027
L-17Xa	~ 3000	3100	3.4	30	4.5

*Probably high because of counting interference from Kr^{85}; the longer half life makes the Xe^{133} results more reliable.

Examination of the interior of capsule L-16b at GEVAL has disclosed almost complete disintegration of the tantalum-sheathed thermocouple and some apparent reaction with the fuel. This result is surprising, since the temperature had not exceeded 2100°F prior to failure of the thermocouple some eight days after the beginning of operation. No similar failures have been observed at such low temperatures. (AEC Activity 4141.1)

Graphite Fabrication. – Investigations of the effect of impregnation of graphite bodies with high-carbon thermosetting furfuryl alcohol solution have continued. Second impregnations of specimens have resulted in increases in final density ranging from 1.50 to 5.63%, compared with the original density. The increase in density due to the second treatment was relatively small in comparison to the increase during the first impregnation. As expected, the increase in density due to impregnation was inversely proportional to the initial density. An autoclave has been fabricated for use in treating the samples to obtain higher densities through impregnation. (AEC Activity 4141.1)

Mechanical Properties of Graphite. – A program for investigating the time-dependent mechanical properties of graphite is being carried out. A stress-strain curve and two relaxation curves have been obtained for specimens of a type of graphite specified for the EGCR core. The relaxation test data demonstrate that the graphite will exhibit time-dependent deformation; however, the data do not indicate which creep laws are followed. Creep tests under constant load and under various loading rates at 750 and 1100°F are scheduled for determining the creep behavior and the time-dependent fracture ductility of the material. (AEC Activity 4141.1)

Dimensional Behavior of EGCR Fuel Capsule at Elevated Temperatures. – The experimental device for studying the dimensional behavior of UO_2 fuel pellets encapsulated in a stainless steel tube to simulate the EGCR fuel element is functioning satisfactorily up to central temperatures of 3000°F in the pellets. Satisfactory radiographic techniques have been developed for measuring both the axial and radial expansion with suitable reproducibility. The first loading of pellets was tested in static helium at maximum central temperatures of approximately 3000°F, with a temperature gradient from the outer surface of the stainless steel tube to the inner surface of the UO_2 that varied up to a maximum of 1000°F. The second loading of pellets is now being tested.

The data obtained thus far indicate that the expansion of the UO_2 is nearly a direct function of the central temperature. However, the contact area between the pellets in a vertical stack, as in the EGCR fuel element, decreases as the radial temperature gradient of the pellet is increased. Where the central UO_2 temperature is in the range of 1800 to 3000°F, the resultant gaps are readily visible on radiographs. At a central temperature of 2270°F, the contact area between pellets is reduced to 80% of the area in contact at room temperature; at a central temperature of 3032°F, the contact area is further reduced to 41%. The total diametrical gap of 0.010 in. that existed in the first loading did not change more than ±0.001 in. (AEC Activity 4141.1)

THERMAL-BREEDER REACTOR PROGRAM

Homogeneous Reactor Program

Homogeneous Reactor Test. – The program of reactor maintenance and alterations which was begun last month continues. So far, six major pieces of radioactive equipment have been replaced or repaired: a core feed pump, a pressurizer purge pump, the core dump valve (removed temporarily), the inlet valves to both high-pressure samplers, and the core sampler mechanism (removed for repair of a valve handle extension).

Concurrently, the core vessel is being altered. The upper five diffuser screens have been cut into 1-1/2-in.-wide strips by means of a remotely operated underwater cutting torch. Approximately 95% of the screen pieces have been removed from the core. (AEC Activity 4151.3)

Homogeneous Reactor Instrumentation. – Life testing was completed on 12 production-model titanium stem-sealing bellows assemblies. These bellows were cycled to destruction in UO_2SO_4 at a temperature of 285°C with

2300 psig differential pressure applied externally. The average life of the 12 bellows was 24,362 cycles. This unit was developed by Fulton Sylphon for a minimum life of 10,000 cycles under the conditions stated. (AEC Activity 4151.1)

Design. – The appearance of a second hole in the HRT core vessel has made it necessary to design additional tools for inspection and patching. To date, tools have been designed to take an impression; to cut, retain, and remove a sample of the core wall metal at the edge of the hole; and to insert a plug.

A revised multiclone assembly installation was designed to achieve higher efficiency in the concentration of solids being fed to the HRT chemical plant. (AEC Activity 4151.1)

Metallurgy. – Studs made from rods of the titanium alloy 6Al-4V (6% aluminum-4% vanadium) produced elliptical failure cross sections when broken in a tensile test. This, as well as the unusual contours of the failure, was typical of rod material. The studs were used in the HRT bellows mockup. After the test, which simulated reactor conditions with the exception of radiation, the bolts were tensile-tested to determine the effects of the exposure on their mechanical properties. It was found that the exposure had little if any significant effect on the properties of the bolts.

The texture (preferred orientation) of sheet specimens of titanium and zirconium alloys gives rise to elliptical tensile failure cross sections, and, in fact, the textures of these materials have been studied by means of these cross sections. The texture produced in rod and wire is usually symmetrical about the rod axis and thus gives rise to circular failure cross sections. The elliptical cross sections of the Ti 6Al-4V bolt failures show that a typical rod texture was not produced in this material. The contour of the failure (cup and cone) was unusual in that the base of the cup was a rough rectangle circumscribed by the ellipse. Again, this indicates a wide divergence from a typical rod texture. These results constitute another striking example of the unusual deformation behavior of the group IVB metals. (AEC Activity 4151.1)

In-Pile Autoclave Solution Corrosion Studies. – A Zircaloy-2 rocking autoclave with Zircaloy-2 specimens was exposed in the LITR for 134 hr at 280°C with a solution of 0.032 m $UO_2(NO_3)_2$, 0.008 m $Cu(NO_3)_2$, and 0.091 m DNO_3 in D_2O and an estimated solution power density of 2.6 w/ml. The purpose of this test was to obtain information concerning the radiation stability of nitrate fuel solutions and the radiation corrosion of Zircaloy-2 in nitrate solution.

Data available from gas pressure measurements, as well as from chemical analyses and specimen weights, show that nitrate ion decomposition occurred during the test. The pressure data also indicate a radiation-induced recombination of the nitrate decomposition products. During the first half of the exposure period a steady-state condition existed between nitrate decomposition and recombination (with about 30% of the nitrate decomposed). Accurate values for the yields of these processes could not be obtained from the pressure data. However, the yield of N_2 was estimated to be as much as a factor of 10 greater than the value (4×10^{-4} molecule per 100 ev absorbed) estimated from results reported by other investigators.

Vapor-phase combination of D_2 and O_2, at efficiencies estimated from results reported by others, roughly accounts for the radiation-induced back reactions. During the last half of the exposure the solution became unstable and the rate of nitrate decomposition increased continuously, possibly because of an effect on the solution produced by excessive stainless steel corrosion in the capillary.

The Zircaloy-2 corrosion rate during the first half of the exposure was estimated to be 6 to 7 mils/year, a value near that expected in a sulfate solution of similar concentration. This rate increased to a maximum of 30 to 35 mils/year during the second half of the exposure. Initially, the yield of D_2 was 1.3 moles per 100 ev, and the rate constant for the copper-catalyzed recombination of D_2 and O_2 was 1600 liters·mole^{-1}·hr^{-1}. Near the end of the exposure, the values for these factors, again at 225°C, were 0.9 and 800. (AEC Activity 4151.1)

Dynamic Blanket Materials Tests. – Procedures have been developed in the thoria pellet test program for evaluating the relative durability of experimental preparations of thoria pellets which might be suitable for use in a proposed pebble-bed blanket of a thermal breeder reactor. The experimental preparations included spheres, right cylinders, and cylinders with hemispherical ends, calcined from 1000 to 1750°C. The spheres were 0.2 in. in diameter and the cylinders were approximately 0.2 in. in diameter by 0.2 in. in length. The preparations were compared in accelerated (1 to 2 hr) tests at room temperature in spouted beds to determine general particle integrity and attrition rates. Static autoclave tests and 300-hr exposures of packed pellet beds in flowing D_2O at 260°C in 100A pump loops were made in order to evaluate high-temperature durability and possible leaching of additives (0.5 to 5% CaO or Al_2O_3 or both) from the pellets.

During the examination of 47 different pellet preparations, weight losses during exposure in D_2O (12 to 200 hr) in static autoclaves at 260°C ranged from below 0.3×10^{-3} to 0.2% per hour. Materials which lost more than 0.3×10^{-3}% per hour failed because of spalling, leaching of additives, or partial disintegration. Sixty-seven per cent of the preparations showed losses below 0.3×10^{-3}% per hour.

Pellets which lost less than 0.3×10^{-3}% per hour in static autoclaves showed weight losses during 1-hr exposures in spouted bed tests (10 pellets, 0.24 fps superficial velocity, 24 fps jet velocity) at room temperature of 0.3 to 30% per hour. Twenty per cent of the materials showed low rates, 0.6% per hour or less.

Two preparations exposed in horizontal packed beds at 2 fps superficial velocity in 100A loop tests at 260°C in D_2O with inert-gas atmosphere gave attrition rates of 0.006 and 0.008% per hour, respectively, during 300-hr exposures. Sixty-five per cent of the lost material was retained as fines in the packed beds. Six per cent of the calcium was leached from one of the preparations which contained 5 wt % CaO as additive. (AEC Activity 4151.1)

In-Pile Slurry Corrosion Loop. – A redesigned core section fabricated of bent piping was incorporated in the prototype 5-gpm in-pile slurry loop. The increased flow velocity (minimum 6 fps) prevented deposition of thoria in the core section during 870 hr of operation at 280°C. The concentration

of the slurry circulated during this test ranged between 450 and 900 g of Th per kg of D_2O; the circulating inventory was in agreement with the book value.

The thoria used in this test contained 1/2 wt % uranium, based on thorium, and was fired at 1500°C. Particle-size degradation from 1.7 to 0.5 μ average particle diameter was noted during the test, but no adverse effect on loop operation was observed.

This out-of-pile test indicates that such a loop is capable of in-pile operation with thoria slurry, and construction of an in-pile loop is in progress. (AEC Activity 4151.1)

In-Pile Autoclave Slurry Corrosion Studies. – Eleven Zircaloy-2 pin specimens from five in-pile slurry autoclave experiments and one unirradiated experiment were analyzed for uptake of hydrogen. The pins were exposed in rocking autoclaves to aqueous slurries of thoria-urania (0.5 or 5% U based on thorium) under added atmospheres of O_2 or D_2, at 280°C, for periods from 314 to 1660 hr, with the development of 252 to 4040 whr/ml of fission energy in the in-pile tests. Originally the pins contained 60, 110, and 120 ppm of hydrogen; the final values were from 120 to 210 ppm, the pickup ranging from 20 to 110 ppm. Little, if any, effect of radiation or atmosphere was evident, but the consistently higher values for tested pins as compared with untested ones implied an uptake due to exposure to high-temperature water. (AEC Activity 4151.1)

Molten-Salt Reactor Program

Chemistry. – A promising procedure for the removal of Pa^{233} from breeder blanket melts, involving precipitation upon the addition of excess BeO, is being studied in the blanket salt $LiF-BeF_2-ThF_4$ (67-18-15 mole %). As confirmed by four separate trials, Pa^{233} (obtained by irradiation of normal thorium) was reduced from initial concentrations corresponding to a gamma-ray activity of 10^5 counts$\cdot min^{-1} \cdot g^{-1}$ to background activity by the addition of 6000 ppm BeO. This same behavior was also obtained in the presence of low dilutions (1000 ppm) of uranium, along with a partial precipitation of uranium. Previous experiments had shown that a larger excess of BeO results in an essentially complete removal of uranium from solution.

Chromic fluoride, an important constituent of the corrosion products which can occur with strongly basic fluoride melts, was found to have a melting point of 1400 ± 25°C. The 1100° melting point currently listed in the literature may have been due to admixture with CrF_2; in the new determinations, disproportionation to CrF_2 and volatile CrF_5 was prevented by the use of sealed platinum capsules.

Graphite degassed at 1300°F in a vacuum for 24 hr incurred the following volume per cent permeations with blanket fluoride melts under 60 psig pressure at 1300°F: GT-123, 0.15; MH4LN-85, 7; and AGOT, 11. Only the results with GT-123 are regarded as satisfactory for molten-salt breeder application. (AEC Activity 4152.1)

Metallurgy. – Two additional alloys for use in brazing graphite to metal have been developed which give promise of having good corrosion resistance to fused salts. These materials, containing the elements Au, Ni,

Mo, Cr, and Fe, were found to wet and flow on fine-grain graphite. Graphite (grade CT-158) specimens were brazed to molybdenum plates without cracking on thermal cycling.

Fuel-permeation tests have been made on three grades of graphite to observe the effect of time on the extent of permeation. Samples of AGOT, R-4, and S-4 grades of graphite were subjected to BULT 14-0.5 U fuel under a pressure of 150 psig at 1300°F for times of 5 and 100 hr. Good agreement between the 5- and 100-hr tests was observed for the values of "bulk volume permeated" for the AGOT specimens and R-4 specimens tested. The S-4 specimens gave anomalous results, in that the short-time test showed higher permeation. (AEC Activity 4152.1)

Corrosion Tests. — Examination has been completed on INOR-8 forced-convection loop 9354-1, which circulated salt 126 for 14,563 hr with a maximum hot-leg temperature of 1300°F. The hot-leg regions showed surface roughening and pitting to a depth of 1-1/2 mils, and metallic deposits in substantial quantities were found in the cold-leg region of the loop. These deposits were composed predominantly of nickel, together with minor amounts of iron and chromium.

The mass transport of nickel observed in this loop necessarily implies that a very powerful oxidant was made available to the salt. An indication of the nature of this oxidant is afforded by analyses of the last salt mixture used, which after test was found to contain a large amount of UO_2. This constituent was most probably produced by H_2O contamination which entered during one or more of the tubing failures encountered during loop operation. Such contamination would also result in formation of HF, which is potentially capable of oxidizing nickel. (AEC Activity 4152.1)

In-Pile Tests. — The first MSR in-pile graphite-fuel capsule experiment, ORNL-MTR-47-1, was terminated on March 10 at the completion of the planned irradiation time. This experiment is scheduled to be sectioned in the MTR hot cells the second week of April, after which it will be shipped to ORNL for examination.

The second experiment is now at the MTR and was inserted on March 28. The designed operating conditions are the same as for the first experiment, namely, fuel temperature approximately 1300°F, fuel pressure 100 psia, and power density about 200 w/cm^3. (AEC Activity 4152.1)

Pump Development. — The molten-salt pump containing a salt-lubricated journal bearing was operated for 105 hr at 1250°F, 1200 rpm, and approximately 50 gpm. During this period the pump rotation was started and stopped 100 times. Upon examination, the salt bearing was found to be in good condition and suitable for further testing.

The MF-F pump endurance test has operated continuously for 23,832 hr, circulating salt 30 at 1200°F. (AEC Activity 4152.1)

Remote Maintenance Demonstration. — All maintenance operations prior to running the system with molten salt at 1300°F have been completed. Filming of a color motion picture for record purposes is in progress. When this is completed, operation at high temperature will commence. (AEC Activity 4152.1)

Thermal-Breeder Evaluation

Thermal-Breeder Evaluation Studies. – The merits of aqueous homogeneous, graphite-moderated molten-salt, and graphite-moderated liquid-bismuth breeder reactors operated at nearly equal fuel-cycle costs ($\sim$1.5 mills/kwhr) were evaluated. The net electrical plant capability was assumed to be 1000 Mw, and the fuel and fertile streams were processed continuously on the site.

The specific powers were 1.2, 1.2, and 0.5 Mw (electrical) per kilogram, respectively, based on fuel, and 5.9, 3.7, and 5.3 Mw (electrical) per ton based on thorium. The net breeding ratios were 1.10, 1.07, and 1.07, giving doubling times of 5-1/2, 11, and 25 years at full power. The fuel-cycle costs at the design points selected were 1.4, 1.3, and 1.6 mills/kwhr.

The aqueous homogeneous reactor has an advantage in breeding ratio and doubling time because D_2O is superior to graphite as a moderator in breeder reactors. The molten-salt reactor has an advantage in fuel-cycle costs and in inventory of uranium in the fertile stream as a result of the use of a solution blanket, which allows economical removal of uranium as it is bred. (AEC Activity 4160)

HIGH FLUX ISOTOPE REACTOR

Critical Experiments. – Detailed neutron flux traverses have been made in the critical assembly supporting the design of the High Flux Isotope Reactor. Small fission chambers, both with and without cadmium covers, were used as detectors. The fluxes of thermal neutrons (i.e., subcadmium neutrons producing fissions) at the center of the fuel region and at the center of the D_2O reflector are about 6 and 29%, respectively, of the flux at the center of the natural water core. The values of the cadmium fraction (fraction of neutrons below cadmium cutoff energy) are 0.99, 0.73, and 0.97 at the centers of the core, of the fuel region, and of the D_2O region respectively. All these results were obtained at the vertical midplane of the critical assembly.

The thermal neutron flux at mid core per unit reactor power has been obtained from experimental data by two methods. The integral of the flux distributions referred to above, together with the thermal-neutron fission cross section, yields a value of 5.4×10^{15} neutrons$\cdot$cm$^{-2}\cdot$sec^{-1} per 100 Mw, the design operating power. A direct measure of the thermal flux with calibrated neutron detectors, together with a measurement, by radiochemical analysis of the U^{235} solution, of the number of fissions resulting in that measured activation of the detectors, gives 5.8×10^{15} per 100 Mw. (AEC Activity 4410)

Heat Transfer. – Burnout safety factors for the HFIR have been calculated to be $\sim$2.7 based on an increase in power level and $\sim$3.7 based on a local hot spot which does not significantly affect the subcooling of the coolant at the burnout location.

Test sections are being fabricated which will be used for studying the influence of spacer ribs on temperature distribution and burnout. Calculations indicate that spacers of width 1/8 in. or less will not seriously affect either the surface temperature or the burnout heat flux.

Further natural-circulation heat transfer tests have established that a highly restricted liquid return path (flow area about one-eighth of the test-section flow area) gives a burnout flux of 60,000 $Btu \cdot hr^{-1} \cdot ft^{-2}$, or 79% as large as that observed with a return path of essentially zero flow resistance. Supplementary natural-circulation burnout tests of the ORR geometry have confirmed that a liquid return path with a flow area considerably less than that of the heated section will give burnout fluxes nearly as large as that with completely unrestricted recirculation.

Final results of the aluminum-plate experiments (without spacers) have been correlated and recommendations made for design. Safe design values may be obtained with the Zenkevich-Subbotin equation (reduced by 30%) for burnout, with the Hausen equation (reduced by 10%) for local nonboiling heat transfer coefficient, and with the Moody plot (increased by 15%) for average friction factor. (AEC Activity 4410)

Corrosion. – Studies concerned with the effect of heat flux on the corrosion of type 1100 aluminum by high-purity (reactor grade) water have continued. To date, five tests varying in duration from three to ten days have been completed, and in all of these tests the conditions have been essentially the same. Water flowed through a cooling channel in an electrically heated aluminum specimen at a rate of 33 fps. Water entered the specimen at 150°F and was heated to 190°F at the exit. The maximum heat flux across the surface of the specimens was 1.6×10^6 $Btu \cdot hr^{-1} \cdot ft^{-2}$.

The results of the five tests have been qualitatively the same; that is, as the test progressed under constant operating conditions the temperature of the aluminum increased. In the ten-day tests the maximum temperature increase has been 225°F.

Only the first test specimen has been completely examined to date. Visual examination of the water-cooled surface showed the surface to be metallic in appearance and essentially unaffected except near the outlet of the specimen, where some "film-stripping" had occurred. Metallographic examination, however, showed that the metallic appearance was deceptive, since all surfaces were covered with a tightly adhering glassy oxide, the thickness of which varied from 0.002 in. near the outlet to about 0.001 in. at the inlet. The formation of this oxide on the surface of the aluminum was responsible for the observed increase in temperature during the test. In the "film-stripped" region (highest aluminum temperature) an irregular white oxide overlay the tightly adhering oxide, and the aluminum had undergone intergranular attack to a depth of 0.005 in. All other surfaces had suffered only slight surface roughening with no evidence of intergranular attack.

Because of the method of fabrication of the test specimen, an accurate determination of the total penetration could not be made. However, based on the results of isothermal tests conducted here, it is estimated that the extent of uniform metal removal would amount to 0.001 to 0.002 in. Thus the extent of penetration was 0.007 in. in the region of highest temperature. (AEC Activity 4410)

Metallurgy. – The inner section of a mark II fuel element has been fabricated for use in a hydrodynamic test. The original design was altered to include (1) anchorage of the fuel plates to the internal tube sheet by

a combination of welding and peening and (2) the insertion of welded comb spacers at both ends of the fuel plates. Measurements have been made of the plates prior to assembly and of the water gap between plates after assembly.

Welding studies have been completed on a method of ruggedizing the attachment of fuel plates to the outer tube sheet of the mark II element. The use of 1/2-in. aluminum bands welded into grooves in the plates, spaced 2 in. apart, appeared to give a rigid joint without visible distortion. This technique will be used to strengthen the fuel assembly fabricated for hydrodynamic testing. (AEC Activity 4410)

Design. – Based on the current HFIR core design, and considering all design tolerances and engineering and calculational uncertainties, the maximum fuel plate surface (oxide-water interface) temperature is estimated to be 362°F, with a maximum hot-spot heat flux of 1.4×10^6 $Btu \cdot hr^{-1} \cdot ft^{-2}$. The minimum burnout heat flux for this reactor geometry is calculated to be a factor of 2.5 above the maximum operating heat flux.

The HFIR specifications call for storage facilities for 27 spent HFIR cores in the storage pool. Shielding requirement calculations were based on a square storage lattice located at the bottom of the pool, with a 2.5-ft minimum spacing between core centers. The maximum dose rate at the pool surface, assuming 14 ft of water between the nearest spent core and the dose point, is calculated to be 2 mr/hr. With a pool side shield consisting of 5 ft of ordinary concrete plus 5 ft of water, the maximum side shield surface dose rate is estimated to be 1.2 mr/hr.

Performance specifications for the reactor building and cooling system, along with drawings of the building and a flowsheet of the cooling system, were completed and released to the architect-engineer. (AEC Activity 4410)

GENERAL REACTOR RESEARCH

Reactor Evaluation Studies. – The fuel value of U^{233} in recycled uranium was calculated relative to a U^{235} value of $17 per gram. To date, calculations have been completed for three complete cycles in five thermal reactors (Yankee, Dresden, Carolinas-Virginia, Hallam, and GCR-II). The value of pure U^{233} varied from $18 to $20 per gram. The value of U^{233} in recycled uranium from an initial U^{233}-thorium cycle was slightly greater than that of pure U^{233} and varied from $20 to $21.5 per gram. The value of U^{233} in recycled uranium from an initial U^{235}-thorium cycle was less than that of pure U^{233} and varied from $10 to $18 per gram. (AEC Activity 4160)

Reactor Safety Studies: BSR-II. – Extensive static and dynamic tests of the BSR-II, the stainless steel–UO_2-fueled alternate core for the Bulk Shielding Facility reactor, have recently been completed in the SPERT-I facility of the National Reactor Testing Station at Arco, Idaho. Data from the tests are being reduced and analyzed.

The self-limiting characteristics of this core were observed in a series of excursion tests to parallel the behavior of the APPR core P/8/19, which had previously been tested at SPERT-I. The self-limiting tests were therefore terminated to prevent excessive core damage. Incipient plate deformation was observed at a peak power of 226 Mw with a period of ~ 14 msec.

Subsequent dynamic testing, utilizing a complete control system, indicated that the system meets and exceeds the design expectations, being capable of controlling excursions with positive periods considerably less than 6 msec.

The reactor and the control system are presently being returned to ORNL. A detailed analysis of the entire series of tests is being prepared. (AEC Activity 4171.1)

Reactor Safety Studies: Reactivity Worth in BSR-I Fuel Loading. — An experimental determination of the reactivity worth of a single Bulk Shielding Reactor-I standard fuel element in the center of a BSR-I loading was completed. This value is of interest from reactor safety considerations, since it determines the initial period by which the reactor power would increase if a fuel element were to be accidentally dropped in an empty position in the center of a critical lattice. The central-element reactivity worth had not been determined previously because the reactivity change produced is too great to be measured by the conventional inhour method. However, the recent installation of a pulsed neutron source (the BSF 300-kv particle accelerator) adjacent to the reactor made the measurement possible.

The experiment was performed on the BSR-I loading No. 78. This loading was delayed critical with all control rods out. The central fuel element (position No. 27) was removed, and the decay period of the prompt-neutron density was measured as 1.29 ± 0.03 msec. From the ratio of the prompt-neutron generation time to the effective delayed-neutron fraction, which had previously been found to be 8.54 msec (±5%), the reactivity worth of the central element was computed to be 5.6 ± 0.3 dollars. (AEC Activity 4171.1)

Fuel Element Catastrophe Studies. — The study of the fission product release from GE-ANP fuel includes a minimum of three melting experiments on each of three types of fuel, as well as a series of extended heating experiments in helium and air at temperatures below the melting point. Results of all these experiments are included in a supplement to ORNL CF-50-1-60.

In the detailed investigation of the fission product rare-gas release from metallic uranium (0.2% burnup) melted in helium, pronounced increases in rate of rare-gas release have been observed on cooling through the freezing point and through the temperature of the α-β transition (665°C). In addition, a momentarily increased rate of gas release has been observed below the latter temperature with additional rapid cooling. These observations suggest that the change in release rate is related to a sudden increase in density. The total magnitude of the three accelerated release effects was quite small (< 1%), since about 98% of the rare gases were released in about 15 min while the uranium was in the molten state. Failure to find a change in gas release rate at the β-γ transition temperature may be due to the small density difference between these two forms of uranium. (AEC Activity 4171.1)

Radiation Detector Development. — A promising application of surface barrier silicon diodes to the problem of neutron spectroscopy is currently under investigation at the Tower Shielding Facility. The application is based upon the $Li^6(n,\alpha)T$ reaction, in which an Li^6 nucleus upon absorption of a neutron emits an alpha particle plus a tritium nucleus in diametrically opposite directions. In the device being tested, a thin layer (~ 150

$\mu g/cm^2$) of Li^6F is vacuum-evaporated upon the surface of one of a pair of silicon diodes, and the coated diode is placed face to face with the uncoated member of the pair in a flux of neutrons. The reactions occurring in the Li^6F coat are simultaneously detected by both members. The energy deposited in the diodes is a function of the neutron energy, thus enabling use of the pair as a neutron spectrometer. Under the experimental arrangement presently being used, a resolution of $\sim 6.5\%$ for thermal neutrons is being observed, reasonably close to the theoretical 5.5% resolution calculated for the present arrangement. (AEC Activity 4410)

Long-Range Planning Studies. — The effective thermal-neutron cross sections and effective resonance integrals for fission products in fluid-fuel reactors were estimated as a function of fuel processing rate. In a U^{235}-fueled reactor having 20% epithermal fissions and a fuel-cycle time corresponding to one fission per fuel atom, the effective thermal cross section was 0.63 times that for unburned fission products; this can be compared with a value of 0.83 for a batch-loaded reactor having an exposure of one fission per initial fuel atom. (AEC Activity 4410)

Nondestructive Test Development. — The program on low-voltage radiography has focused on direct comparisons of required exposures in air and helium atmospheres through a range of x-ray energies. This has been done on bare film and is being done with different absorber thicknesses of aluminum. Further work will include other materials. From this work tabulated data will be available to predict the condition under which a helium atmosphere will be advisable.

The study of ultrasonic behavior in thin sections requires very close control of the ultrasonic frequency. For this reason it is planned to use the pulser of the ultrasonic attenuation comparator, since it can provide continuously variable frequencies from less than 1 Mc to 200 Mc. Calibration of the pulser has begun. Included in the calibration is a measurement of the frequency response of the wide-banded piezoelectric transducers which are available.

In the continuation of the program on gamma assay of U^{235} core blanks, samples of 7 and 13 wt % U^{235} have been received. The data obtained from the analyses of these core blanks will be used to calibrate the spectrometry equipment.

Efforts on problem materials have been primarily on small-diameter beryllium tubing. The very high ultrasonic velocity (approximately twice that in conventional metals) and the low radiation absorption are prime inspection difficulties. Adequate radiographic techniques have been developed, and successful wall-thickness measurements by resonance ultrasonics have been made. Further work is necessary on the pulse-echo ultrasonic technique and the eddy-current method for flaw detection. An eddy-current technique for flaw detection has been developed for large graphite cylinders and is presently undergoing evaluation.

In continuation of the program to develop inspection standards, a variety of arc-discharge machined notches have been produced in tubing to simulate cracklike defects. Efforts are presently in progress to calibrate the machining equipment so that notches about 0.0025 in. deep can be produced with an accuracy of ±0.0001 in. At present the equipment is capable of an accuracy of only ±0.0005 in. (AEC Activity 4420)

Mechanical Properties Research. — Irradiation of graphite can produce either contraction or expansion, depending on the temperature of the graphite. Such volume changes in a structure under restraint can readily produce stresses in excess of the ultimate strength of the material. Metals are able to relieve thermal stresses and those imposed by phase changes through plastic flow. Unless graphite is able to relax stresses in a similar manner severe design limitations will be imposed on its use in power reactors. Tensile creep tests and relaxation properties of AGOT-type graphite are being evaluated from ambient to 1100°F. Results at 750°F indicate that a Newtonian viscosity exists, so that the creep rate varies linearly with the applied stress. The value of the viscosity was calculated to be 8.75×10^{10} poises. The rupture stress and the elongation at rupture have not demonstrated strain-rate or time dependency.

Stress-rupture tests of type 304 stainless steel have been run in environments of argon containing various partial pressures of oxygen. An environment of argon containing 2 ppm oxygen decreases the creep rate as compared with that observed in pure argon. The creep rate is increased as the oxygen concentration is increased, and the specimens tested in pure oxygen exhibit a higher creep rate than those tested in pure argon. These results indicate that two competing processes are occurring; the material is being strengthened by the solution of oxygen and weakened by the penetration of oxygen along the grain boundaries. Low oxygen levels would tend to favor the first process, whereas higher oxygen levels would contribute significantly to the second process. (AEC Activity 4420)

Fuel Cycle Development. — Bulk densities of ThO_2 and mixed ThO_2-UO_2 as high as 9.0 g/cc were obtained by vibratory compaction in vertical 3/8-in.-OD, 0.035-in.-wall stainless steel tubes. The materials used were arc-fused ThO_2 with a particle density of 9.98 g/cc and arc-fused ThO_2 containing 3.4% UO_2 of particle density 10.0 g/cc. Densities were highest when a blend of three sized fractions of the fused oxides, 58% 10 to 16 mesh, 15% 70 to 100 mesh, and 27% -200 mesh, was vibrated with a pneumatic piston-type vibrator at basic frequencies of 40 to 80 cps and accelerations below 40g in tubes clamped at the base only. Vibration instrumentation mounted at the top of the tube registered complex vibrations with steady accelerations of about 80g and unstable peaks of 300g.

The particle density of a ThO_2 powder containing 5% UO_2, prepared from coprecipitated thorium uranous oxalates, was 10.2 g/cc, and that of a similar composition prepared by adsorption of uranyl ammonium carbonate onto 650°C-fired ThO_2 was 9.99 g/cc as measured pycnometrically with toluene. These oxides were fabricated by pelletizing the 650°C-prefired precipitates, firing to 1650°C for the first preparation and to 1750°C for the second, crushing, and screening. (AEC Activity 4420)

Power Reactor Fuel Processing: Engineering Studies. — Use of a typical flowsheet for Yankee Atomic fuel gave maximum flow capacities of compound extraction-scrub pulsed columns of 1130, 1060, and 1410 $gal \cdot ft^{-2} \cdot hr^{-1}$ (flow rate in extraction section) for sieve, nozzle, and nozzle-zebra cartridges respectively. Flooding rates, usually limited by the scrub section, decreased to about 50% of maximum with increasing pulse frequency from 30 to 70 cpm. In strip columns, maximum flow capacities were 1100 and 1530

$gal \cdot ft^{-2} \cdot hr^{-1}$ for sieve and nozzle cartridges respectively, and flow capacities decreased with increasing pulse frequency from 25 to 50 cpm. Although the flow capacities of both extraction and strip columns were approximately the same, production capacity is limited by the strip column, because under flowsheet conditions the ratio of combined flow rates in the latter to those in the former is nearly 2.0.

HETS values at a pulse frequency of 50 cpm for uranium extraction were 2.0 ft in the sieve plate column with a top interface and 4.0 ft with a bottom interface. The HETS in the nozzle plate columns (bottom interface) was 2.0 ft with 0.125-in.-dia holes and 3.0 ft with 0.188-in.-dia holes. HETS values at 35 cpm for uranium stripping were 2.9, 2.1, and 6.3 ft, respectively, for 0.125-in.-dia-hole sieve plates and 0.125- and 0.188-in.-dia-hole nozzle plates with a top interface; the values were 2.4, 2.0, and 4.5 ft, respectively, with a bottom interface. (AEC Activity 4441)

Power Reactor Fuel Processing: Darex Process. — A literature survey of the thermodynamics and limits of flammability of gases expected to be generated during the Darex (and Sulfex) decladding of stainless-steel-clad NaK-bonded fuels indicated that vapor-phase explosions are a definite possibility with the Darex (5 M HNO_3–2 M HCl) system when used with NaK-bonded fuels. Reactions between nitrogen oxides and hydrogen are highly exothermic, and mixtures of the gases are explosive over wide concentration ranges.

The hydrogen content of Darex off-gases from dissolution of stainless-steel-clad oxides is very low. Measurements show that the hydrogen content of the off-gas from batch dissolution of type 304 stainless steel increased with increasing HCl concentration for several HNO_3-HCl concentrations. The average increase was from 0.14% with 5 M HNO_3–2 M HCl to 2.5% with 1 M HNO_3–6 M HCl; the maximum concentration from the latter dissolvent was 4% for 30% metal dissolution.

The solubility of sintered UO_2–0.8% fissia ceramic pellets in Darex and Sulfex dissolvents was studied. Insolubles ranged from 1 to 9% of the fissia material, mainly Zr, Mo, and Ru; much larger quantities of silica from the stainless steel jackets were present.

The presence of 0.1 M borate in initial Thorex dissolver solution lowered the corrosion rate of titanium in the solution phase from 6–7 to 0.3 mil/month. However, the borate did not eliminate pitting in the vapor space above solutions with high thorium concentrations. Specimens of type 304L stainless steel that were exposed to Darex-Purex simulated waste solutions continued to show intergranular attack in solutions at 65 and 85°C; however, over-all rates for approximately 2000 hr exposure were only 0.1 and 0.41 mil/month respectively. (AEC Activity 4441)

Power Reactor Fuel Processing: Sulfex Process. — Consolidated Edison type fuel pellets (96% ThO_2–4% UO_2; 92% of theoretical density) were exposed in the ORR to power densities of 16 to 160 Mw per metric ton of thorium for 3 min. In no case were the fuel pellets shattered.

Four successive semicontinuous Sulfex decladding and Thorex core dissolution cycles were run in engineering-scale equipment on unirradiated prototype Consolidated Edison fuel assemblies to determine reaction rates, dissolution rates, decladding time, product loading, cross contamination of the clad and core dissolvents and its effect on passivation, and thorium and uranium losses to the decladding solution. The results of the first

two runs showed average decladding rates of 1.64 and 2.27 $mg \cdot cm^{-2} \cdot min^{-1}$ with 660 and 530% excess boiling 4 M H_2SO_4; instantaneous rates varied from 2 to 51 g/min. Clad solution metal loading varied inversely with dissolvent flow rate and ranged from 35 to 27 g of stainless steel per liter. From 2.78 to 4 hr was required for decladding. The average nitrate concentration in the clad solution was approximately 0.004 M; the sulfate concentration in the core solution varied from 0.001 to 0.006 M. Instantaneous nitrate concentrations in the clad solution varied from an initial value of 0.015 M to a final value of 0.0005 M. Seven to nine hours was required to dissolve approximately 90% of the core. The core dissolvent was initially 13 M HNO_3–0.04 M F^-–0.1 M Al^{+++} and was recycled at average flow rates of 0.6 and 1.0 liter/min. Thorium and uranium losses to the decladding solution varied from 0.022 and 0.017% in the absence of a heel to 0.08 and 0.1% in the presence of a 10% heel. (AEC Activity 4441)

Power Reactor Fuel Processing: Alternative Aqueous Processes. – Since the dissolution rate of ThO_2-UO_2 in Thorex dissolver solution (13 M HNO_3–0.04 M F^-–0.1 M Al^{+++}) is relatively slow, alternative dissolvents are being sought. Fused sodium or potassium hydroxides at temperatures of ≥ 400°C were used to shatter prototype Consolidated Edison pellets of 93% of theoretical density. Most of the resulting fragments had diameters less than the radius of the original pellet. Similar pellets were dissolved in refluxing 14.8 and 7.4 M H_3PO_4 at rates of 14 and 0.08 $mg \cdot cm^{-2} \cdot min^{-1}$, respectively, and in fused ammonium bifluoride at 220°C at a rate of 7 $mg \cdot cm^{-2} \cdot min^{-1}$.

A method was developed for simultaneously disintegrating to -20 mesh and leaching the uranium from 1.2 × 1.4 × 4.5 cm pieces of graphitized fuels by boiling in fuming nitric acid (90% HNO_3, 0.2% maximum dissolved oxides). Two successive acid treatments followed by water-washing recovered not less than 99.85% of the uranium in fuels containing ≥ 5% uranium; recovery decreased to 99% when the uranium concentration in the fuel was only 2%. When 70% acid was used instead of 90% in the second leach, metal recoveries decreased by at least 0.2%. (AEC Activity 4441)

Power Reactor Fuel Processing: Zircex Process. – When nickel turnings were used as de-entrainment packing for the uranium chloride condenser, > 10% of the uranium was lost to the zirconium chloride condensate.

In scouting tests for a material of construction for N_2-CCl_4 vapor at 600°C, ten alloys were tested. Illium R, Nichrome V, Inconel, and INOR-8 performed best, though minimum rates in every case were ≥ 26 mils/month. (AEC Activity 4441)

Power Reactor Fuel Processing: Equipment and Material Evaluation. – A simple pneumatic density probe tested in water slurries of sand (105–150 μ) gave readings consistently 3 to 6% higher than calculated for a homogeneous mixture. Detection of solids by this method appears feasible except in the case of highly hydrated silica (3% SiO_2, 97% H_2O), for which the observed density was equal to the density of water.

In tests to evaluate protective coatings for use in a deionized water-filled fuel storage canal where exposure of 3×10^9 r at 135°F is anticipated, Amercoat No. 74 surfacer with No. 66 seal coat, an epoxy formulation, appeared undamaged by exposure to 2×10^9 r. Phenoline-300, a modified

phenolic, and Ceilcrete-6400, a polyester, failed in deionized water at 1.7×10^9 r and 4×10^8 r respectively. However, Phenoline-300 appears undamaged but is darkened after gamma exposure of 3×10^9 r in air. (AEC Activity 4441)

Power Reactor Fuel Processing: Mechanical Processing. — Mechanical equipment installed in the high-level Segmenting Facility to demonstrate the dejacketing of stainless-steel-jacketed NaK-bonded SRE core 1 fuel operated satisfactorily in initial mechanical operability tests.

During extended operation of the Syntron vibrating-spiral conveyer-leacher with 1/2-in.-dia by 1-in.-long stainless steel slugs, two severe blockages occurred across the 4-in.-wide spiral flights. This may limit the usefulness of this equipment. (AEC Activity 4441)

Power Reactor Fuel Processing: Solvent Extraction Studies. — Uranium and thorium were simultaneously stripped from a neutral 30% tributyl phosphate (TBP) solution by an equal volume of 0.008 M $Al(NO_3)_3$ in seven stages. A greater volume of strip solution or more stages are required to strip the uranium and thorium from organic solutions containing > 0.1 M HNO_3. Distribution coefficients of uranium and thorium were determined for a system containing 30% TBP—Amsco, 0.2 to 20 g/liter of uranium, 0 to 70 g/liter of thorium, and no acid. (AEC Activity 4441)

Power Reactor Fuel Processing: Zirflex Process. — Tin was dissolved at rates exceeding 10 $mg \cdot cm^{-2} \cdot min^{-1}$ in 5 M HF–2 M H_2O_2 at 13 to 70°C. The rate at 13°C was < 10% of that in 5 M NH_4F–2 M H_2O_2.

In Zirflex dissolver solutions the presence of 0.003 to 0.03 M H_2O_2 lowered the corrosion rates of Hastelloy F, type 309SCb stainless steel, and Ni-o-nel by factors varying from 5 to 10. In the presence of 0.03 M H_2O_2, maximum rates for a 9-hr exposure were 1.2, 1.7, and 2.2 mils/month for Hastelloy F, type 309SCb, and Ni-o-nel respectively. In Zirflex solutions and fluoboric—nitric acid solutions the presence of 0.4 to 0.6 M Cr(VI) lowered the corrosion rate of titanium from 500 to 40–60 mils/month. Titanium—0.2% Pd alloy showed somewhat higher corrosion rates than Ti-45A in these high-fluoride solutions. (AEC Activity 4441)

Fluoride Volatility Processing. — The dissolution of niobium, stainless steel, and thoria by hydrofluorination in fused fluorides was investigated for possible applications of the volatility process. In the presence of HF, niobium metal dissolved at rates of 1 to 3 $mg \cdot cm^{-2} \cdot min^{-1}$ in 31-24-45 mole % $LiF-NaF-ZrF_4$ at 600°C. As with zirconium, the niobium coupons were covered with deposits, presumably metallic, from reducible impurities in the melt. Dissolution rates of type 347 stainless steel were 0.4, 0.8, and 1.2 $mg \cdot cm^{-2} \cdot min^{-1}$ in 60-40 mole % $NaF-ZrF_4$ at 500, 600, and 650°C respectively. In one test with 42.0-46.5-11.5 mole % KF-LiF-NaF the stainless steel dissolution rate was about 4 $mg \cdot cm^{-2} \cdot min^{-1}$ at 600°C.

Thoria pellets (1750°C-fired) reacted relatively slowly with gaseous HF in a 57-43 mole % LiF-NaF melt, at a dissolution rate of only 0.3 $mg \cdot cm^{-2} \cdot min^{-1}$ at 700°C. The same pellets and arc-melted thoria dissolved completely in 5 to 10 min in 31-24-45 mole % $LiF-NaF-ZrF_4$ at 650°C without the use of HF. The resulting oxyfluoride solution was hydrofluorinated or directly fluorinated rapidly. In one run, thorium concentrations were 17.8,

14.2, and 12.3 wt %, respectively, in filtrates from dissolution mixtures made at 700, 650, and 600°C.

Molybdenum, at a concentration of several thousand ppm, was the major contaminant in the UF_6 product of the volatility process in laboratory-scale tests with "Nautilus" fuel. Although MoF_6 behaved similarly to UF_6 in the NaF absorption-desorption process, the similarity does not appear to be so great that they cannot be separated. Preliminary data from a chemical investigation of the MoF_6-NaF system indicated that the complex is $MoF_6 \cdot 3NaF$, and that the equilibrium partial pressure at 25°C is > 1 mm, while that for UF_6 is $\sim 10^{-6}$ mm in the NaF-UF_6 system.

All work in the Volatility Pilot Plant was suspended on November 20, 1959, as a result of contamination from an adjacent facility. In the ensuing cleanup, portions of the installed equipment were removed. Review of the equipment design indicated the need for modifications. The redesign, which is essentially complete, alleviates some of the possible hazards. (AEC Activity 4442)

PROGRAM 5000 — PHYSICAL RESEARCH

Reactor Operations. — Preparatory work for the annealing of the Graphite Reactor, scheduled for this summer, will require that the reactor be shut down for one to two weeks beginning about the last of March.

The ORR has operated with only a small amount of trouble. In the last two cycles the reactor has operated 98.2% of the time.

Laboratory Facilities. — Various containment jobs are being designed or studied. These include long-term improvements to the radioactive waste systems, liquid and gaseous waste monitoring, filters in the cell ventilation system, emergency storage for process waste, demineralizers for the Graphite Reactor canal, and containment for the Segmenting Cell and Graphite Reactor buildings. Since the design and installation of the final monitoring systems will take some time, temporary monitoring is being set up wherever possible, particularly on the stacks.

PHYSICS

Neutron Diffraction. — Neutron diffraction investigations have been performed on a powder sample of TbSb to determine the existence of magnetic ordering in this compound. Antiferromagnetic reflections were observed below about 17°K, and these reflections can be indexed on the basis of a magnetic unit cell in which all three lattice vectors of the cubic, rock salt type chemical unit cell are doubled. The intensities are in accord with a magnetic structure of the MnO type in which ferromagnetic (111) planes of moments are oppositely directed in alternate layers. The observed atomic magnetic moments of $8.2 \pm 0.8\ \mu_B$ are aligned parallel to the [111] direction. (AEC Activity 5220)

Scintillation Spectrometry and Instrument Development. — Gamma radiations emitted in the decay of 2.3-hr I^{132} have been investigated with single-crystal and coincident scintillation spectrometers. The energies and relative intensities of the gamma rays observed are 0.24(1), 0.515(17), 0.64(22), 0.673(78), 0.775(65), 0.953(15), 1.142(3), 1.299(2), 1.392(6), 1.45(1), 1.75(0.1), 1.92(0.7), 1.99(0.6), and 2.40(0.1) Mev. The data are consistent with levels in Xe^{132} at 0.673 ± 0.009, 1.448 ± 0.012, 1.964 ± 0.015, 2.10 ± 0.03, 2.401 ± 0.016, and 2.84 ± 0.02 Mev. There is also some evidence for levels at 2.59, 2.75, and 3.24 Mev. This decay scheme is similar to that proposed by Finston and Bernstein [Phys. Rev. 96, 71 (1954)]. From the values obtained for the comparative half lives of the beta-ray transitions and the ground-state spin of I^{132} ($X_0 = 4$) [Bull. Am. Phys. Soc. 4, 386 (1959)], the higher-energy levels of Xe^{132} are each expected to have a spin of 3, 4, or 5. Of these spins, the results of gamma-gamma angular correlation measurements and the locations of transitions in the proposed decay scheme are in best agreement with the assignments 4, 3, 4, 3, and 3 or 5 for the 1.448-, 1.964-, 2.10-, 2.401-, and 2.84-Mev levels respectively. (AEC Activity 5220)

Fast-Chopper Time-of-Flight Spectrometer. — Additional measurements have been made on the total neutron cross section of B^{10} from 0.018 to

0.04 ev with samples enriched to 99.89 and 96.39% in B^{10}. The total cross section of B^{10} in this energy range follows the relation $\sigma_T(\text{barns}) = (612 \pm 6)/\sqrt{E(\text{ev})}$. The total cross section of B^{10} for neutrons of energy 0.0253 ev (velocity = 2200 m/sec) is 3850 ± 40 barns. (AEC Activity 5220)

High-Voltage Experimental Program: (α,n) Cross Sections. — Thick-target (α,n) neutron yields have been measured from threshold to an alpha-particle energy of about 11 Mev for isotopically enriched targets of nickel, copper, and zinc. The absolute neutron yields, which were measured at approximately 100-kev energy intervals, have an accuracy of ±4%. The neutron detector was the 4π, flat-energy-response graphite sphere detector developed by Macklin [Phys. Rev. 114, 571 (1959]. Cross sections for the (α,n) reaction were obtained to an accuracy of ±15% by differentiation of the smooth thick-target yield curves. Values for the measured cross sections varied from 50 μb for Cu^{65} near threshold to over 100 mb for most of the nuclei at an alpha-particle energy of 10 Mev. For those nuclei in which the (α,n) cross section is thought to account for most of the reaction cross section, the observed cross sections are considerably larger than those predicted by Shapiro [Phys. Rev. 50, 171 (1953)] for his larger radial parameter of $1.5A^{1/3} \times 10^{-13}$ cm. Both the shape and the absolute values of the observed cross sections agree well with the recent optical-model analysis of reaction cross sections for alpha particles by Igo [Phys. Rev. 115, 1665 (1959)]. (AEC Activity 5220)

High-Voltage Experimental Program: Angular Distribution of Fission Fragments. — Detailed measurements were made of the fission cross section of U^{234} from threshold to a neutron energy of 4 Mev. A minor dip was observed at 330 kev, and a major peak at 850 kev, followed by a minimum at 1030 kev. Fragment angular distribution (0°/90°) ratios were taken at several points from 400 kev to 3.7 Mev, with particular attention being paid to the regions around the latter two extrema, where fragment distributions at angles of 30 and 60° were also measured. The curve of fission fragment asymmetry is seen to bear a striking resemblance in shape to that for the total fission cross section.

Similar angular distribution measurements were made for U^{235}, with particular attention to the region around 900 kev, where a sudden jump is known to exist in the total fission cross section. No effect on the angular distribution was found in this region, however. (AEC Activity 5220)

CHEMISTRY

Raw Materials Research and Development. — A promising method has been developed for the recovery of thorium from primary amine—hydrocarbon solvents. Thorium is stripped from the solvent with 2 M NaCl—0.5 M Na_2SO_4, and from this solution more than 90% of the thorium is precipitated by heating in a continuous precipitator for 2 hr at 60°C. The precipitate settles and filters rapidly. The residual thorium in the filtrate is sufficiently low to allow recycle of the filtrate to the stripping system. (AEC Activity 5310)

Solvent Extraction Research. — Further studies of the distribution of rare earths and americium between tributyl phosphate (TBP) and 9.9 M LiCl–0.1 M HCl gave coefficients in the order Ce:Eu:Y:Am:Lu ≈ 1:2:2:7:35. A multistage extraction experiment with americium and europium confirmed the usefulness of this system for separation of the transplutonic elements from all the fission product lanthanons. Extraction with the system TBP–12 M HNO_3 separates the transplutonic elements only from the lanthanons lighter than neodymium.

In continued study of the aggregation behavior of the amine extractants, light-scattering measurements were extended to solvents other than benzene. In n-nonane containing 3% tridecanol, the observed aggregation numbers were 2 for di(1-isobutyl-3,5-dimethylhexyl)amine sulfate, 6 for 1-(3-ethylpentyl)-4-ethyloctylamine sulfate, and 5 to 12 for di-n-decylamine sulfate. The scattering ratios for this last solute showed a smooth curvature corresponding to a continuous change in the average molecular weight from 3300 at 0.005 g/ml to 8100 at 0.036 g/ml.

A nitrite-acetone treatment of the aqueous feeds considerably improved the decontamination from both zirconium-niobium and ruthenium in the Purex codecontamination cycle. In this treatment a dissolver solution of about 2 M uranium and -0.3 M HNO_3 (acid deficient) is adjusted to 0.03 M $NaNO_2$ and digested at room temperature for 1 hr; after subsequent adjustment to +0.3 M HNO_3 (free acidity), 1 vol % acetone is added, and, after 1 hr of heating at 90°C, the solution is adjusted to Purex aqueous feed conditions. Countercurrent tests with TBP and di-sec-butyl phenylphosphonate (DSBPP) as extractants showed that this feed treatment increases the decontamination factors for both ruthenium and zirconium-niobium by ten- to fortyfold. The decontamination factors for ruthenium were 1.3×10^4 and 1.1×10^4 with 1 M solutions of TBP and DSBPP, respectively, in Solvesso 100. The corresponding decontamination factors for zirconium-niobium were 1.2×10^4 and 2.3×10^4. (AEC Activity 5310)

Extraction Reagent Performance. — To test its stability toward radiation damage, six samples of 1 M di-sec-butyl phenylphosphonate (DSBPP) in Solvesso 100 were contacted with 1 or 0.3 M HNO_3 and then irradiated to levels of 45, 135, and 400 whr/liter with 1-Mev electrons. For solutions contacted with 1 M HNO_3, the nitric acid extracted by the solvent was destroyed with yields decreasing from 2.88 to 0.97 molecules per 100 ev absorbed as the dose was increased from 45 to 400 whr/liter. The corresponding values for solutions contacted with 0.3 M HNO_3 were 0.63 and 0.17. A monobasic acid was formed by decomposition of the DSBPP. For solutions contacted with 1 M HNO_3, the yield of monobasic acid decreased from 0.79 to 0.20 as the dose was increased from 45 to 400 whr/liter. The corresponding values for solutions contacted with 0.3 M HNO_3 were 0.21 and 0.13. (AEC Activity 5310)

Ion Exchange Technology. — A chelating resin, Dowex A-1, removed strontium from simulated neutralized Purex waste to which tartrate had been added to prevent iron precipitation. Its strontium capacity was significantly higher at pH 7.5 than at 5.5, and at both pH's the capacity for rare earths was much lower than that for strontium. Both strontium and rare earths were readily eluted with 2 M HNO_3. The capacity for both strontium and rare

earths was lower than that of a sulfonated cation exchange resin, Dowex 50, but elution from Dowex A-1 was easier and separation of strontium from rare earths was better.

Sulfur determination indicated sulfonic acid capacities of 4.5 meq per dry gram for unirradiated Dowex 50 resin and 3.0 meq per dry gram for resin irradiated to almost 12 whr per dry gram. Titration of these resins indicated strong-acid capacities of 4.8 and 4.7 meq per dry gram respectively. The reason for the difference between the two analytical methods is being investigated. It could mean that irradiation separated sulfonic groups from the resin structure but also added other acid groups by oxidation or some other mechanism.

More than 1000 volumes of ORNL low-level process water waste, containing added Cs^{137} and Sr^{85} and adjusted to pH 11.6, was passed through Duolite C-3 sulfonated phenolic resin to give an effluent containing less than 0.1% of the influent cesium and strontium.

The rate of uranium loading from 0.005 M UO_2SO_4 that was 0.025 M in total sulfate was determined for nitrate-equilibrated samples of Dowex 21K sized to 1200, 960, and 820 μ. The rates corresponded approximately to apparent uranium diffusion coefficients of 1.5×10^{-7}, 0.90×10^{-7}, and 0.90×10^{-7} cm^2/sec respectively. Elution of all these sizes with 1 M $NaNO_3$–0.1 M HNO_3 gave rates corresponding to apparent uranium diffusion coefficients of approximately 1.6×10^{-7} cm^2/sec. Preliminary data indicated that the rate of uranium loading on sulfate-equilibrated 1250-μ Permutit SKB corresponds approximately to an apparent uranium diffusion coefficient of $\sim 4.9 \times 10^{-8}$ cm^2/sec. The value for Dowex 21K of the same size is notably higher, $\sim 1.2 \times 10^{-7}$ cm^2/sec. Uranium was eluted from Permutit SKB by nitrate at a rate approximately the same as that from Dowex 21K (diffusion coefficient 1.6×10^{-7} cm^2/sec). (AEC Activity 5310)

Physical Chemistry of Molten Salts. – Studies of the phase equilibria in the binary system CsF-UF_4 have disclosed that the compound $CsF \cdot 6UF_4$ occurs as an equilibrium phase. The compound $CsF \cdot 6UF_4$ melts incongruently to UF_4 and liquid at 863°C. It is uniaxial negative with refractive indices $N_\omega = 1.598$ and $N_\epsilon = 1.590$. This compound is isostructural with $RbF \cdot 6UF_4$. (AEC Activity 5330)

High-Temperature Aqueous Solution Chemistry. – The effect of added metal ions on the decomposition of peroxide in simulated reactor fuel solution based on uranyl nitrate was studied from 60 to 100°C. The effects of adding small amounts of iron, copper, chromium, and nickel to the solution in the form of dissolved salts were determined. The iron acted as a very effective homogeneous catalyst, the rate of decomposition increasing by a factor of 20 for each ppm of iron in solution. The addition of copper to an iron-containing solution enhanced the observed catalysis; this enhancement reached a maximum value at about 30 ppm of copper, which increased the observed rate of decomposition by about a factor of 3; little change was observed as the copper was further increased. In solutions containing both iron and copper, the observed rate was not clearly first order with respect to peroxide, in contrast with the first-order dependence usually noted in previous studies of peroxide decomposition. Neither nickel nor chromium had a similar effect. Copper, nickel, and chromium, either singly or in combination, had little catalytic effect. The addition of any combination of these metal ions had no observable effect on the temperature dependence

of the decomposition process. All these results are consistent with results previously obtained in the sulfate system. (AEC Activity 5330)

Chemical Separation of Isotopes. – Spectral data obtained from molecules containing N^{14} and N^{15} have been used to calculate very accurate partition function ratios at temperatures from 100 to 600°K for the following ions and molecules: N_2, N_2O, NO, NOCl, NO_2, NO_2^-, NO_3^-, and N_2O_4. Corrections for anharmonicity were made whenever the necessary data were available.

One hundred fourteen grams of N^{15} (purity 95% or better) was obtained from the operation of the Nitrox system from December 1, 1959, to March 1, 1960. (AEC Activity 5340)

Separation of Oxygen Isotopes. – Oxygen isotopes were fractionated by equilibrating oxygen gas with the solid bis-disalicylalethylenediimine complex of cobalt. Oxygen-18 was enriched in the gas phase, and a single-stage separation factor of 1.012 ± 0.002 (95% confidence interval) was obtained. (AEC Activity 5340)

METALLURGY AND SOLID STATE PHYSICS

High-Temperature Reactions. – Procedures have been developed for analyzing the ultraviolet absorption spectrum of NO_3^- into component bands. These procedures consist in fitting 50 to 100 data points to theoretical models of the spectral profile with a least-squares criterion. This spectrum consists of a weak band ($n \rightarrow \pi^*$ transition) overlapped by the edge of a strong band. The analysis yields the intensity-dependent parameters of the weak band and specifies the adequacy with which the theoretical models represent the composite spectrum. These procedures have been applied to the spectra of pure, molten alkali nitrates. Some results are as follows: The band shape is closely approximated by the Lowry-Hudson profile with one exception. Cesium nitrate has a distinct Lorentzian wing and, hence, would be approximated better by the Voigt profile. However, the contribution of this wing to the oscillator strength is small. Oscillator strengths range from 0.85×10^{-4} for rubidium nitrate to 4.0×10^{-4} for lithium nitrate. Thermal broadening is appreciable but is compensated in such a way that the oscillator strength increases by only a small amount with increasing temperature. The Kuhn-Braun profile, which is commonly assumed in band-shape analysis, gives an oscillator strength which is about 6% too small. (AEC Activity 5420)

THERMONUCLEAR PROJECT

Cross Section Studies. – The dissociation cross section of H_2^+ by hydrogen has been measured for energies of 200 through 600 kev. An H_2^+ beam incident upon a differentially pumped hydrogen gas target was electrostatically analyzed for H_2^+ and its dissociation products H^0 and H^+. Detection was made by means of a Faraday cup and a secondary electron collector. Both the total-loss cross section for H_2^+ and the proton-production cross section are in close agreement with the previously reported results for energies of 200 through 500 kev and with results for energies of 500 through 600 kev. (AEC Activity 5540)

DCX-1 Facility. – Preliminary measurements have been made on the energy of the escaping neutral particles from the trapped plasma. With an electrostatic energy analyzer, the energy spectrum of particles escaping from the midplane exhibits a maximum at 300 kev. At 1-1/4 in. Z-wise from the midplane the energy spectrum peaks at 210 kev. Additional studies will be made in order to determine in more detail the energy loss of the trapped protons.

During March, DCX was moved to Building 9201-2. Coincident with the move, additional vacuum pumps are being installed to provide sufficient pumping speeds with the gas arcs. Operation of DCX will probably be resumed by April 15. (AEC Activity 5540)

DCX-EPA. – The burned-out coils have been replaced by coils of the DCX type. These coils have been tested, and the machine is ready for experimental use. (AEC Activity 5540)

Ion Source Development. – Ion currents up to 300 ma at 80 kv have been obtained from a Von Ardenne type of source. This current was obtained from a newly designed source which has improved cooling on the intermediate electrode and the anode. Calorimetric measurements of the extracted ion currents have been made and agree within 3% with the Faraday-cup measurements. A system for analyzing high-current beams (30 to 500 ma) at 60 to 100 kv has been built and is now being installed.

All components for the 600-kv-beam test facility have been completed. Assembly is in progress and should be finished by April 15. (AEC Activity 5540)

Theory. – A very general set of Fokker-Planck equations for particles and radiation was previously obtained. These have now been solved for the case of an isotropic uniform plasma with no external fields. The plasma particles exhibit additional friction forces due to interaction via transverse waves. The corresponding oscillator equation shows the expected dispersion relations.

The steady-state Fokker-Planck code is in operation and is being applied to several simple energy degradation problems. (AEC Activity 5540)

PROGRAM 6000 – BIOLOGY AND MEDICINE

BIOLOGY

Insect Cytology and Genetics. – The effects of low doses of radiation can be readily detected in the rapidly and continuously dividing neuroblasts of the grasshopper embryo. The mitotic rate of these cells is temporarily reduced 75% by 3 r and 25% by 1 r of x rays. Chromosome fragments are found in 2% of the neuroblasts after exposure to 3 r of x rays; this represents breakage of 0.1% of the total number of chromosomes treated (males have 23 and females 24 chromosomes in neuroblasts). Exposure to 1 r of x rays induces chromosome fragments in 1.4% of the cells treated; 0.06% of the total number of chromosomes irradiated are broken by 1 r. Chromosome fragments have never been observed in unirradiated neuroblasts. (AEC Activity 6130)

Pathology and Physiology. — Male mice of the RF strain were exposed when 5 to 6 weeks of age to whole-body 250-kvp x radiation at a rate of 80 to 100 r/min (about 100 mice per group), as follows: group I, 0 r (control); group II, 25 r; group III, 50 r; group IV, 75 r; and group V, 100 r. The incidence of myeloid leukemia in the respective groups was: I, 4%; II, 6%; III, 10%; IV, 10%; and V, 17%. The median survival time (in months) of the groups was: I, 19; II, 19; III, 19; IV, 19; and V, 18. The incidence of lymphomas did not differ significantly from one group to another. These results are consistent with earlier observations suggesting that the dose for doubling the incidence of myeloid leukemia in the RF mouse is less than 100 r. However, further study will be required in order to determine whether the dose-response curve for this disease is or is not linear within the dose range 25 to 100 r. (AEC Activity 6130)

Biophysics: Magnetic Resonance. — The production of electron spin resonances in *Tradescantia* pollen has been found to require the dissipation of about 190 ev per resonance for 300-kvp x rays. The decay of these resonances is being followed, and preliminary analysis shows an initially rapid decay followed by a prolonged, almost steady-state resonance pattern. Preliminary studies indicate that this initial rapid decay can be increased by exposure to ultraviolet radiation subsequent to the treatment with x rays. (AEC Activity 6130)

Biophysics: Ultraviolet Studies. — Continued studies of the production of chromosome breakage in *Tradescantia* pollen by ultraviolet radiation and x rays have shown definitely a large synergistic action for these agents. The effect has been found for both pre- and posttreatments with ultraviolet radiation. It appears to be almost independent of the interval between treatments up to periods of 2 hr. The magnitude of the synergistic effect is very large; for example, a small dose of ultraviolet, sufficient to give a barely perceptible aberration frequency (~ 2 or 3%) if applied alone, when given before or after a moderate x-ray dose (150 rads) leads to an aberration frequency up to four times that for strict additivity. Additional experiments have indicated a very drastic dependence of ultraviolet-induced aberration on the moisture content of the pollen during irradiation. (AEC Activity 6130)

Radiation Protection: Living Cells. — The resistance of *Escherichia coli* to inactivation by x rays has been found to be remarkably influenced by the preirradiation culture conditions. As was reported previously, the resistance is well correlated with changes in the hydrogen ion concentration produced as a result of the metabolic activity of the growing cells in aerated cultures. The lower the final pH attained over the range 8.4 to 4.8, the more resistant the resulting cell populations. The changes in resistance to x-ray inactivation are paralleled by changes in resistance to ultraviolet and heat inactivation. Resistance or sensitivity to inactivation is not an effect of intracellular or extracellular pH per se but seems to result from differences in the metabolic activity or products accumulated in the cell during the course of growth. Attempts to demonstrate an extra- or intracellular metabolic product of resistant cells that could act as a chemical protective agent have been unsuccessful. Since the resistance is not restricted to damage by ionizing radiation, this possibility seems somewhat remote.

Several alternative explanations for the development of resistance are being tested, some of which depend on the ability to demonstrate the gain or loss of resistance by cells with incubation for relatively short periods of time before irradiation. Preliminary experiments indicate that partial loss of resistance can be measured within an hour of incubation in media favorable for growth; also, some data suggest that cell division may be necessary for a complete change in cell population from resistant to sensitive type. (AEC Activity 6230)

Mammalian Radiation Recovery. — The killing effect of isologous or homologous blood in irradiated mice which have been treated with homologous bone marrow can be prevented by x irradiation of the blood, either in vitro or in vivo. This suggests that there is a nucleated cell with the capacity to divide and become immunologically active which normally circulates in the blood of the mouse.

Investigation of the ability of amino acids to protect mouse bone marrow cells against damage by freezing and thawing has shown that, while most are at least somewhat protective, those with a free SH group and those with two amino groups are not. The nonprotective compounds tested include, in the first class, L-cysteine and mercaptoethylguanidine; and in the second class, L-asparagine, L-lysine, and L-arginine.

The bone marrow cells of Chinese hamsters given 100 r of whole-body irradiation have been fixed, stained, and scored for chromosome aberrations at various time intervals after irradiation. The total chromosome breakage value fell from 50% at 2 hr to 19% at 6 hr, 17% at 10 hr, 11% at 12 hr, 4% at 18 hr, and 2% at 30 hr. The control breakage figure was 0%.

Mice bearing ascites tumor cells were exposed to x-ray doses between 600 and 5000 r seven days after inoculation. Cell diameters were measured at daily intervals after exposure. The data indicate abnormal cell enlargement similar to that found in intestinal crypt cells after irradiation. These data also show that enlargement may be a dose-dependent phenomenon, since greater enlargement was observed after higher doses of x rays. (AEC Activity 6230)

Radiation Immunology. — Preliminary studies on the relative radiosensitivity of antibody-forming cells from normal and preimmunized donors showed that no significant difference exists between these cells. It was found that there are 2×10^7 spleenlike cells per gram of mouse.

In an attempt to determine the cellular distribution and antigenic potency of homologous hematopoietic transplantation antigens, a new model is being studied which involves an assay for antibody-forming cells. The data suggest that the minimum single dose of bone marrow cells which will sensitize a mouse to reject 50% of the test cells is less than 10^4. In contrast, a previous model which involved death as an end point required 10^8 cells or more for a questionable result.

Determination of the antibody-forming capacity of donor and recipient cells in radiation-induced rat-mouse chimeras 30 days after treatment with radiation and bone marrow revealed that spleen cells of chimeras failed to synthesize antibodies against mouse red blood cells when transplanted into heavily x-irradiated rats. On the other hand, these cells synthesized antibodies against rat red blood cells when transplanted into heavily x-irradiated mice. These results show that, since no titratable antibodies can

be found in the chimeras, there exists a state of paralysis in these rat-mouse chimeras, and that the competent cells are those of the recipient.

The diffusion chamber technique makes possible the study of cell differentiation, somatic mutation, relations between cell maturation and protein synthesis, interaction of cells with foreign material, etc. Diffusion chamber studies are valid provided the cells do not migrate through the pores. Millipore membranes with pore size 0.45 to 1.2 μ have been customarily used in the past. In mice, however, recipient hematopoietic cells (10 to 15 μ in diameter) do enter these chambers by diapedesis when the pore sizes are 0.45 and 1.2 μ, but not when the pore size is 0.1 μ. (AEC Activity 6230)

PROGRAM 8000 — WORK FOR OTHERS

International Reactor Program at ORNL. — Twenty-three students currently enrolled in the foreign programs have completed 21 weeks of school since the beginning of classes in November. The 13 Operations Supervision students will complete their class work at ORSORT in April and will then begin full-time participation for three months in the Laboratory's reactor operations. ORSORT and Hazards Evaluation students will continue classes until the beginning of study projects in August.

Plans have been made for starting a third session of foreign programs this fall. Descriptive material for these programs is being prepared for distribution.

Three hazards evaluation reports and four operations supervision reports, compiled by students of the 1959 session, have been published this month. These reports summarize the results of study projects following class work and student laboratory assignments. (AEC Activity 8717)

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

March 1960

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