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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

MARCH 1958

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March 1958

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

TABLE OF CONTENTS

	Page
PROGRAM 4000 — REACTOR DEVELOPMENT	6
HOMOGENEOUS REACTOR PROJECT	6
Homogeneous Reactor Test	6
Mathematics and Computation: Homogeneous Reactor Development	7
Reactor Analysis	7
Homogeneous Reactor Instrumentation	8
HRP Design	8
HRP Radiation Damage	8
Small-Scale Dynamic Slurry Corrosion Tests	9
Dynamic Slurry Loop Corrosion Tests	9
In-Pile Autoclave Slurry Corrosion Studies: LITR	9
CIVILIAN-POWER ADVANCED GAS-COOLED REACTOR (GCR)	10
Gas-Cooled Reactor: Design	10
Gas-Cooled Reactor: Experimental Engineering	11
Gas-Cooled Reactor: Radiation Effects	11
Gas-Cooled Reactor: Metallurgy	11
GENERAL REACTOR RESEARCH	12
Metallurgical Materials and Processing	12
Fuel Element Catastrophe Studies	12
Reactor Safety Studies	13
Long-Range Planning Studies	13
PROGRAM 5000 — PHYSICAL RESEARCH	13
Reactor Operations	13
ORR Project	14
PHYSICS	14
Neutron Velocity Selector	14
High Voltage Program	14
Neutron Diffraction	15
Thermal Diffusion	15
CHEMISTRY	16
Raw Materials Chemistry	16
Thermochemistry and Calorimetry	16
Chemistry of Corrosion	16
Ion Exchange Studies	16

[redacted]

[redacted]

TABLE OF CONTENTS (Continued)

Page

Program 5000, Continued:

Chemistry of Technetium 17
Characterization of Short-Lived Fission Products 18
High-Temperature Aqueous Solution Chemistry 18
Chemical Separation of Isotopes 19

METALLURGY 19
High-Temperature Reactions of Metals and Ceramics 19

PROGRAM 6000 — BIOLOGY AND MEDICINE 20

BIOLOGY 20
Pathology and Physiology 20
Biophysics 20
Microbial Protection and Recovery 20
Mammalian Recovery 21
Radiation Immunology 21

PROGRAM 8000 — WORK FOR OTHERS 22
Oak Ridge School of Reactor Technology 22

MISCELLANEOUS REPORTS 23
Open Literature Publications 23
Visitors 25

[redacted]

[redacted]

OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

March 1958

This Status and Progress Report summarizes somewhat more than one-half the activities of the Laboratory. Some of the topics are included every month, but many of them are reported on a bimonthly schedule. Program 4700 is covered in a separate report.

PROGRAM 4000 — REACTOR DEVELOPMENT

HOMOGENEOUS REACTOR PROJECT

Homogeneous Reactor Test. — Replacement of the faulty in-cell electrical wiring with copper-jacketed cable was completed on March 15. Concurrent with this activity, a new set of corrosion specimens was inserted into line 103, the fuel circulating pump was reinstalled, and the fuel letdown valve was replaced with a new unit by means of underwater and dry maintenance techniques. The cover flange over the core thimble was removed prior to flooding the cell, and a standpipe was attached, to permit internal examination of the core without lowering the shield water level. An external radiation survey of the accessible portions of the fuel system with a waterproofed probe indicated a fairly uniform gamma activity of 15 to 30 r/hr outside the piping, with the exception of two hot spots of 500 and 1100 r/hr, respectively, in the horizontal connecting line to the fuel pressurizer and at the sediment pot at the foot of the fuel dump tanks.

On March 15, the interiors of line 100 and the core were examined with periscopes; an isolated mound of solids in a location roughly corresponding to the hot spot described above was observed in line 100, and a copious accumulation of similar material around the perimeter of the topmost diffuser screen was noted in the core. Samples of the core accumulation were subsequently recovered for analysis by vacuuming them into a filter; material was obtained from line 100 with a scraper on a flexible shaft. The wall thickness of line 100 was checked with an ultrasonic probe on March 20, after which the shield water level was lowered and the thimble cover flange replaced. A corrosion sample is now attached to the underside of the cover flange, to investigate the effect of impingement of the fuel solution at the core exit.

The reactor cell was resealed on March 21, and it passed a leak test at 15 psig. The startup preparations, including the addition of a new charge of fuel, are in progress for the next power run. (AEC Activity 4103.3)

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Mathematics and Computation: Homogeneous Reactor Development. — The ratio of thermal flux at the center of a reactor to the power generated in the fuel region was calculated for a number of reactors on the basis of a three-region, two-group model. Survey-type calculations were performed on the Oracle and the IBM 704. Cylindrical and spherical geometries were considered; fuel was assumed to be in the annular region, with moderator in the other two regions.

A machine program was written for calculating the stresses arising in the transition joint between Zircaloy-2 and stainless steel in HRE-3 type reactors. A nonuniform fuel-burnout calculation for a heterogeneous-type slab reactor was programed. The calculation gives fuel exposure in Mwd/ton as a function of fuel concentration and enrichment and calculates the spacial distribution of U^{235}, Pu^{239}, and fission-product poisons. (AEC Activity 4103.1)

Reactor Analysis. — A thermal analysis was completed for the HRT dome, in which the maximum wall temperature was estimated by assuming reactor operation at 10 Mw and considering the outer dome wall to be either insulated or uninsulated. The results indicated that the maximum wall temperature may exceed 320°C with the dome insulated. With the dome uninsulated, the maximum wall temperature was estimated to be less than 300°C.

The decrease in HRE-3 breeding ratio due to the presence of a Zircaloy core tank and shroud was examined as a function of metal thickness and spacing. For the case of a 1/2-in.-thick core vessel, and a 1/2-in.-thick shroud spaced 5 in. from the core tank, the breeding ratio would be 0.047 less than if there were no Zircaloy present. For the spacing given above, more than 50% of the blanket power would be released within the region between the core tank and shroud.

The possibility of criticality occurring in the HRE-3 slurry dump tanks was examined by estimating the critical concentration for an infinite system and comparing it with the fuel concentrations which might occur if the core fuel were mixed with the blanket material. If uniform mixing occurs, criticality cannot be attained. All the core uranium inventory would have to be added to just one-fifth the thorium present in the blanket in order to attain criticality at 20°C.

The nuclear characteristics of some spherical double-blanket reactors were studied. The core was considered to be 4 ft in diameter, the pressure vessel was 9 ft in diameter, while the blanket was divided into two regions; the region adjacent to the core was assumed to be either 6 in. or 1 ft thick. If the annular blanket region contained 4000 g of Th per liter and the outer blanket contained 500 g of Th per liter, negligible reactivity effects would occur if slurry settling took place in the dilute outer blanket region. (AEC Activity 4103.1)

Homogeneous Reactor Instrumentation. — The capacity of the capillary pressure-drop system used to measure the flow of oxygen into the HRT high-pressure fuel system was increased by replacing the original capillary with a shorter length (2 ft) of 0.019-in.-ID tubing. The capacity of the new system is 2600 std cc/min, with a pressure drop across the thermal valve and capillary of 300 psi (2100 to 1800 psi). The thermal-valve temperature was 285°C for this flow.

A control analysis of the HRT heat exchanger feed-water system was made which indicates that the present system is capable of smooth control in the power range from 1 to 5 Mw. To achieve higher power operation, a larger size trim will be needed in the feed-water valve to the blanket heat exchanger.

Waterproof differential-transformer position transmitters were installed on the HRT letdown valves. These position transmitters will provide some information on the feed-pump flow rate, gas letdown rate, and pressurizer-level control stability in the reactor fuel and blanket systems.

Coordination of the design, procurement, and installation of a new power wiring system for use inside the HRT reactor containment tank was completed. The new system, consisting of magnesium oxide—insulated wires in copper tube sheaths, replaces the Fiberglas-insulated wires sealed in conduits, which were subject to breakdown when moist.

A capacitance probe with an associated measurement system for indicating liquid level in a 2500-psi system was tested at low temperature and exhibited a negligible change in capacity when thorium oxide slurry was introduced into the test pot. (AEC Activity 4103.1)

HRP Design. — A special plug was designed to permit replacement of the wiring to the HRT circulating pumps, the space-cooler motors, and the pressurizer heaters, with a minimum of interference with the existing system and components.

Preliminary studies were made, and conceptual layouts were prepared for HRE-3 main heat exchangers, reactor vessel, core and blanket pressurizing-system components, and core and blanket dump tanks. Studies were made in order to determine the activity of slurry following an extended period of reactor operation, the feasibility of using the blanket heat exchanger as a feed-water heater rather than as a steam generator, and the feasibility of letdown from the blanket high-pressure system through a long pipe rather than by means of a valve. (AEC Activity 4103.1)

HRP Radiation Damage. — The extent of annealing of radiation effects at 575°F in three pressure-vessel steels was studied at 5×10^{18} fast neutrons/cm^2. Comparison samples were simultaneously irradiated at 200°F. The transition-temperature shifts ranged from 95 to 165°F at the lower temperature and from 10 to 105°F at the higher temperature of irradiation in Charpy V-notch specimens. It is concluded that the higher irradiation temperature is beneficial in reducing the magnitude of the radiation effects, but there is

at least a 5:1 variation between different steels in the benefits so obtained. The steel with the smallest increase in transition temperature at 200°F (type ASTM A-212 grade B, 6 in. thick, fine grained) showed the least reduction in radiation effects during irradiation at 575°F. The structural or compositional factors causing the differences in the response to the irradiation temperature are not known. (AEC Activity 4103.1)

Small-Scale Dynamic Slurry Corrosion Tests. — In toroid screening tests at 100°C of materials of interest for possible application in slurry feed pumps and circulating pumps, corrected attack rates at 20 fps of <0.2 mil/year for Elgiloy (Rockwell hardness E-36), 1 mil/year for heat-treated Elgiloy (Rockwell hardness C-48), 18 mils/year for Kennametal K-501, <0.02 mil/year for Rexalloy A-33, and <0.2 mil/year for Kearfott A-97 sintered Al_2O_3 were observed in runs with oxygenated slurries of 1600°C calcined ThO_2 at a circulating concentration of 500 g of Th per kg of water.

In toroid tests made in order to ascertain the stability of three experimental sols containing ~400 g of Th and 200 g of SiO_2 per liter, the preparations were flocculated after circulation at 26 fps for 300 hr at 250 and 280°C. The average attack rates (corrected) of the pin specimens exposed in the tests were: type 347 stainless steel, 0.6 mil/year; titanium-75A, 0.6 mil/year; and Zircaloy-3A, 2 mils/year. (AEC Activity 4103.1)

Dynamic Slurry Loop Corrosion Tests. — A system attack rate of 2 mils/year was observed for a 100A loop test of 1003 hr duration made at 280°C and employing an oxygenated slurry of 1600°C-calcined, classified thoria of 1.5 μ mean particle size at an average circulating concentration of 1480 g of Th per kg of water. Pits approaching 100 mils in depth were found on the pump impeller rim where the velocity was 90 fps. The average attack rates of pin corrosion specimens exposed during the run at relative velocities of 21 and 42 fps, respectively, were: austenitic stainless steels, 2 and 23 mils/year; titanium alloys, 1.5 and 6 mils/year; and Zircaloy-2, 0.1 and 1 mil/year. The attack at the higher velocity was due primarily to erosion.

In a second test made at 280°C, with an oxygenated slurry of 1600°C calcined thoria which was classified to provide a mean particle size of 0.9 μ, a loosely adherent deposit of thoria formed in the system pressurizer and necessitated stopping the run after 94 hr of circulation at an average concentration of 870 g of Th per kg of water. The average attack rates of pin specimens exposed in the test at relative velocities of 20 and 40 fps, respectively, were: austenitic stainless steels, 4 and 16 mils/year; titanium alloys, 5 and 8.5 mils/year; and Zircaloy-2, 0.8 and 1.3 mils/year. The system attack rate for the test was 3 mils/year. (AEC Activity 4103.1)

In-Pile Autoclave Slurry Corrosion Studies: LITR. — An average corrosion rate of 0.8 mil/year, based on 1070 hr at 280°C, was observed for a Zircaloy-2 autoclave containing an oxygenated thoria-urania slurry in D_2O. It was exposed for a total irradiation time equivalent to 690 hr of LITR operation at 3 Mw in the HB-6 rocking autoclave at an estimated flux of

5×10^{12} neutrons·cm^{-2}·sec^{-1}. The corrosion rate, which was based on the oxygen consumption deduced from pressure measurements, was 1.7 mils/year for the first week of exposure. The slurry contained 993 g of Th per kg of D_2O and 0.021 m MoO_3 and was prepared from thoria containing 0.5 at. % uranium (93% enriched) based on thorium.

The recombination of radiolytic gas was generally consistent with first-order kinetics, the rate at 280°C being 0.12 mole of D_2 per liter per hour at a D_2 partial pressure of 24 psi. No significant decrease in the recombination activity of the MoO_3 was noted. (AEC Activity 4103.1)

CIVILIAN-POWER ADVANCED GAS-COOLED REACTOR (GCR)

Gas-Cooled Reactor: Design. — Upon the completion of the GCR-2 design report, an evaluation was made of the effects of changes in the steam turbine selected for the plant. It was found that a $3,000,000 cost saving could be obtained by using a 3600-rpm turbine rather than an 1800-rpm unit. A comparison was also made of the Kaiser and ORNL designs and the associated costs. As a result of this study and discussions with both Kaiser and AEC representatives, the ORNL basic costs were validated, and reductions were made in the Kaiser costs. The inherently more expensive installation envisioned by the Kaiser design was responsible for costs approximately 25% higher than those for the ORNL design.

Preliminary estimates were made of the effects of replacing the stainless steel capsules of the GCR-2 fuel elements with graphite. It was found that, with a fuel enrichment of about 1.5% and graphite capsules, the multiplication factor would be the same as with the 2% enriched fuel in stainless steel capsules. The conversion ratio in the graphite-clad system is 0.85, as compared with 0.74 for the more highly enriched system. It was estimated that the higher conversion ratio increases the reactivity lifetime by about 10%, reduces the dollar investment in uranium by 33%, and decreases the fuel burnup cost by about 0.2 mill/kwhr if no credit is taken for the increased burnup that could be achieved.

A study of the economics of power production by a small graphite-moderated reactor operating at a high gas pressure was partially completed. Heat transfer analyses of 1500-psia UO_2-helium systems produced a family of fuel element configurations with which the temperature limits of 1200°F cladding temperature and 4600°F central temperature could be met. Two configurations were chosen for physics calculations; each consisted of a 19-rod cluster, and UO_2 slug diameters of 0.318 and 0.409 in. were considered. The stainless steel cladding thickness was taken as 0.018 in. The infinite multiplication factor, leakage, conversion ratio, and reactivity lifetimes were calculated for a range of lattice spacings and fuel enrichments with a core height of 10 ft and core diameters from 8 to 16 ft. The data are currently being used to establish the fuel cycle costs for this family of small-core reactors. (AEC Activity 4119)

Gas-Cooled Reactor: Experimental Engineering. — In the program of tests of the compatibility of graphite and various metals of interest for fuel element cladding in a helium atmosphere at 1400°F, two vessels, one containing TSF graphite and the other AGOT graphite, were cooled and opened for examination after having been heated for 1000 hr. Visual inspection revealed no apparent damage to 300-series stainless steels, but there was moderate to extensive damage to zirconium and its alloys. Metallographic examination will be required to determine the effects, if any, on samples of tantalum, molybdenum, and various types of carbon and stainless steel. The graphite showed no effects other than a slight discoloration that appeared to be associated with the zirconium specimens. (AEC Activity 4119)

Gas-Cooled Reactor: Radiation Effects. — Specimens of 15% enriched UO_2, which will generate a power density of ~750 w/cm^3 and will operate at a UO_2 surface temperature of 1600°F, were inserted into the LITR. These specimens are being irradiated primarily to obtain fission-gas-release data, but approximately the same thermal stresses as those anticipated in a GCR fuel element will be present, and therefore information should also be obtained on the integrity of UO_2 under GCR conditions. A preliminary design was completed for the modification of a hot cell as required for examinations of irradiated UO_2 fuel specimens. (AEC Activity 4119)

Gas-Cooled Reactor: Metallurgy. — Fabrication procedures were developed for composites of molybdenum and tungsten with additions of silver and MoS_2 which are being considered for use as high-temperature (1400°F) bearing materials. The fabrication procedure consists in cold-compacting and sintering at 900°C, followed by hot-pressing at temperatures near the melting point of the MoS_2 (~1300°C).

Tests of thin-walled tubes to determine their creep-buckling characteristics were continued. A 500-hr test was completed on 3/4-in.-dia type 316 stainless steel tubes with 20-mil walls. The test temperature was 1675°F, and the external pressure was 300 psi. Diametral clearances between the tube wall and the inserts which simulated UO_2 slugs were 6, 9, 14, and 24 mils. Buckling occurred to some degree in all the specimens. The specimen with the 24-mil clearance buckled in a two-lobe mode. The specimen with the 14-mil clearance buckled in a one-lobe mode the full length of the specimen. The 9-mil-gap specimen also buckled in a one-lobe mode, but only over about three-fourths the length of the specimen. The 6-mil-gap specimen buckled only over one-third its length. Previous tests of Inconel specimens had indicated even collapse of the tubes around the inserts, rather than buckling, when the diametral clearance was 10 mils or less.

A 20-mil-wall, 3/4-in.-dia type 304 stainless steel specimen without an insert, which was subjected to an external pressure of 300 psi at 1500°F, was found to be collapsed when removed from the test chamber at the end of 65 hr. Cracks were observed where the tube collapsed around the end plugs, but complete rupture through the wall did not occur.

Gas samples obtained at every 100°F drop in temperature during cooling of the vessels in the graphite-metal-helium compatibility tests described above showed noticeable increases in impurity concentrations soon after the cooling was started. It is postulated that at the test temperature of 1400°F the gaseous impurities evolved by the graphite are rapidly consumed by the active metals contained in the pot, and thus the impurity concentrations in the gas during the test are low. During cooling, however, the rate of consumption becomes less than the rate of evolution, and a net buildup of impurities in the gas occurs.

An encircling-coil eddy-current instrument is being fabricated for the inspection of GCR capsule tubing. The instrument is designed specifically for the inspection of 3/4-in.-dia, 0.020-in.-wall stainless steel tubing. It is expected that this instrument will detect inner and outer surface cracks as shallow as 0.001 to 0.002 in. in the 0.020-in. wall. Preliminary experiments also indicate that an immersed-ultrasonic technique can be applied to the inspection of the closure welds for the GCR fuel capsules. (AEC Activity 4119)

GENERAL REACTOR RESEARCH

Metallurgical Materials and Processing. — Magnesium diffuses into zirconium and Zircaloy-2 predominantly along the grain boundaries at temperatures below the melting point of magnesium. A metallurgical process, based on the characteristic grain boundary diffusion, would destroy the integrity of zirconium and Zircaloy-2 cladding, and thus assist chemical methods for recovery of unburned uranium from reactor fuel elements.

It was established that magnesium diffuses through 0.03-in.-thick zirconium and Zircaloy-2 after 3 hr at 625°C, generating a brittle grain boundary phase. The susceptibility of the grain boundary material to selective attack was tested by exposure to the following acid solutions: concentrated HNO_3, 5 M HNO_3—2 M HCl, 6 M HCl, 4 M H_2SO_4, and 0.04 M HF—13 M HNO_3. Significant attack was observed in the last solution only, as judged by weight changes and metallographic examination. The solution attack was general rather than intergranular, and weight changes observed for magnesium-diffused specimens were the same as for ordinary zirconium and Zircaloy-2. It was concluded that the grain boundary material was not sensitive to attack by the various acid solutions mentioned. (AEC Activity 4203)

Fuel Element Catastrophe Studies. — The products released by the melting of a reactor fuel element were fractionated by 5- and 0.8-μ (mean pore diameter) Millipore sieves in series in order to determine the distribution of the fission products between vapor and particulate matter. The experiment was inconclusive, since all the activity released was found to be Cs^{137}, and all of it was stopped by the 5-μ filter. The use of radioautography for particle size determination has also been unsuccessful. The fuel element used in this study was of the APPR type, had been irradiated to 15% burnup, and had cooled for more than two years, hence only long-lived fission products were involved. (AEC Activity 4252)

Reactor Safety Studies. — The design of a tank which will be used to study the effect of voids in the reflector of a swimming-pool reactor is near completion. The tank will contain two layers of water-filled Plexiglas boxes, 17 boxes in each layer. Adjacent boxes in the two layers can be separated so that voids will extend through the reflector to the reactor face. The size of the voids will vary from 2 in. in cross section to the size that will give a 2-1/2% reactivity effect. A distributed poison method of measuring the reactivity effect will be used. This method employs B_4C-impregnated Plexiglas strips, which are inserted throughout the reactor core. Preparations for the experiment include several calculations for predicting the effect of the voids. (AEC Activity 4252)

Long-Range Planning Studies. — The maximum fuel temperature for the proposed enriched ORNL Graphite Reactor has been calculated; reactor operation at 7 Mw has been assumed, and various methods of fuel loading have been considered. With zero burnup of the fuel, the maximum fuel temperature was 800°F. If 40% burnup of the fuel was obtained by inserting the fuel into the reactor through one face and removing it from the opposite face, the maximum fuel temperature was 950°F. With fuel inserted into the reactor through both faces and effectively removed from the reactor center after 40% burnup, the maximum fuel temperature was 950°F. If alternate channels of the reactor were fed with fuel through one face, and the remaining channels were fed with fuel from the opposite face, the maximum fuel temperature was 850°F if 40% fuel burnup was obtained.

The ratio of thermal flux at the center of a reactor to the power generated in an annular fuel region was calculated on the basis of a three-region, two-group model. Spherical geometry was assumed, and either H_2O or D_2O was considered as the moderator. The moderator was in all three regions, while the fuel (U^{235}) was restricted to the annular region. The maximum thermal flux per megawatt for the D_2O-D_2O-D_2O system ranged from 8.1×10^{13} to 9.8×10^{13} neutrons·cm^{-2}·sec^{-1} if the central-region radius was between 0 and 20 cm and the fuel annulus thickness was between 2 and 30 cm. For the H_2O-H_2O-D_2O system the maximum flux per megawatt was greater than 2×10^{14} for a central-region radius of 5 cm and an annular thickness of 3 cm. (AEC Activity 4510)

PROGRAM 5000 — PHYSICAL RESEARCH

Reactor Operations. — The Graphite Reactor stored energy has been measured at ORNL, the Bureau of Standards, and Brookhaven National Laboratory, and has been found to have a maximum value of 32 cal/g to 250°C. Tests are under way to determine whether the stored energy can be successfully released by external heating with hot air applied through tubes in individual channels.

Radioactivity monitors are being placed on the Building 3019, Isotope Area, and Graphite Reactor stacks. These will be monitored in the Graphite Reactor control room.

ORR Project. — Modifications to the ORR building by the H. K. Ferguson Company have been completed, and ORNL forces are now installing hot cell windows, manipulators, and similar equipment.

The reactor became critical for the first time at 6:06 PM on March 21, 1958, with a critical mass of approximately 1280 g of U^{235}. Work to date has been confined to checkout of instruments and operators before starting on the program of control-rod calibrations and flux measurements.

Tests on the building accident containment features are in progress and should be completed within the next few days.

The 350-kw diesel-powered generator has been installed and is now being tested.

PHYSICS

Neutron Velocity Selector. — Since the installation of the digital speed control the M-1 chopper has been routinely operated at speeds up to 6000 rpm. With a 45-m flight path and a 2.6-μsec neutron burst (at 6000 rpm) a resolution of 0.06 μsec/m is realized. The equipment was used to measure the total cross sections of silver, gold, tungsten, and iodine in the energy range 20 to 400 ev. Resonance parameters, level spacings, and strength functions ($\overline{\Gamma}_n^o/D$) were obtained for gold and iodine. Measurements of the scattering cross section were made for thin tungsten and silver targets in the energy range 20 to 150 ev. When these scattering measurements are compared with the measurements of the total cross sections, assignments of the spins of the compound states (0 or 1 for these nuclides) can be made. The spin states of the levels corresponding to the resonances at 27.1, 46.6, 48.1, and 102 ev in W^{183} were determined by using the scattering of the two resonances at 21.2 and 117 ev in W^{182} (for which g = 1) for calibration. Since the resonances at 27.1 and 46.6 ev have neutron widths greater than their radiation widths, it is possible to determine the spins of these states from thick- and thin-sample measurements of the total cross section. These total-cross-section results agree with those from the scattering measurements. The results from scattering measurements on silver for the resonances at 52, 57, and 72 ev agree with previous assignments. (AEC Activity 5220)

High Voltage Program. — By use of the doubly charged alpha-particle beam from the ORNL 5.5-Mv Van de Graaff and the graphite sphere neutron detector described by Macklin (Nuclear Instr. 1, 335, 1957), total, absolute neutron yields have been measured for the following reactions, the alpha-particle energy range (Mev) covered being indicated in parentheses: $Li^7(\alpha,n)B^{10}$, B^{10*}(4.4—8.2); $Be^9(\alpha,n)C^{12}$, C^{12*}(2.5—8.2); and $B^{10}(\alpha,n)N^{13}$, N^{13*}(2.5—4.8). Major resonance energies observed for the compound nuclei formed are: for B^{11}, 5.15 (11.95), 7.15 (13.22); for C^{13}, 2.58 (12.44), 4.00 (13.43), 4.50 (13.78), 5.75 (14.64), 7.8 (16.06); for N^{14}, 2.99 (13.75), 4.7 (14.97); where the first number given is the alpha-particle energy (Mev) and the number in parentheses is the corresponding excitation energy (Mev) of the compound nucleus.

The reaction $B^{10}(n,\alpha)Li^7$ has been computed by reciprocity for energies up to $E_\alpha = 5.64$ Mev, the threshold for $Li^7(\alpha,n)B^{10*}$. The cross-section extrapolation to zero neutron energy is in good agreement with directly measured values. The cross section for $Li^7(\alpha,n)B^{10*}$ has been computed by using literature values for the cross section of $B^{10}(n,\alpha)Li^7$ and the reciprocity relation.

Cross sections for (p,n) reactions have been measured with an accuracy of $\pm 10\%$ from 2.5 to 5.5 Mev for four isotopes of palladium, five of cadmium, and four of tellurium. For each isotope the cross section increases strongly and smoothly with proton energy: from ~0.02 mb at 2.5 Mev to 53 mb at 5.5 Mev for the palladium isotopes, from ~0.01 to 47 mb for the cadmium isotopes, and from ~0.01 to 28 mb for the tellurium isotopes. At a given energy the isotopes of one element have cross sections differing from each other by only ~20% over most of the energy range, and in each of the three cases there is a definite tendency for the heavier isotopes of a given element to have the higher cross sections. These striking regularities, which are in sharp disagreement with the erratic behavior found by Blaser et al. (Helv. Phys. Acta 24, 3, 1951), indicate that the predominant isotope effect on these cross sections is a simple reduction of Coulomb barrier with increasing mass number and, therefore, with increasing radius. (AEC Activity 5220)

Neutron Diffraction. — Neutron diffraction observations were made on MnI_2 at temperatures down to 1.35°K to determine the existence of antiferromagnetic ordering in this compound at low temperatures. Since MnI_2 crystallizes in the hexagonal layer structure characteristic of CdI_2, it is isomorphous with $MnBr_2$, which becomes antiferromagnetic at 2.16°K (Phys. Rev., in press), but the interatomic distances in the iodide are about 10% larger than those in the bromide. Antiferromagnetic reflections were observed from MnI_2 below 3.40°K, and the ordering transition to the antiferromagnetic state is extremely sharp. The magnetic structure could not be determined from powder diffraction patterns, and therefore single-crystal investigations were made with the specimen in an external magnetic field. These experiments showed that the antiferromagnetic ordering consists of a structure domain pattern similar to that found in $MnBr_2$ but with a different arrangement of the atomic magnetic moments. When an external magnetic field is applied to the sample, the entire crystal is transformed into a single antiferromagnetic domain, and this condition is only slightly altered when the magnetic field is removed. (AEC Activity 5220)

Thermal Diffusion. — Separation of the isotopes of krypton is still in progress. Approximately 35 std ml of Kr^{86} at a concentration of 99.22% is contained in the plant. This should reach the desired concentration of 99.5+% in about two weeks, after which time material of this analysis should be produced at a rate of 100 std ml per week for use in wavelength-standard discharge tubes. (AEC Activity 5250)

CHEMISTRY

Raw Materials Chemistry. — The degree of aggregation in an organic solution of amine salts and extracted metal complexes is important in determining the extraction mechanism. Light-scattering measurements (ORNL-2355), confirmed in several cases by isopiestic measurements, showed that the sulfates of some, but not all, amines are aggregated in benzene solution. 1-(3-Ethylpentyl)-4-ethyloctylamine has an aggregation number of ~10; di-n-decylamine, ~40; bis(1-isobutyl-3,5-dimethylhexyl)amine, 4; methyldi-n-octylamine, 8; and tri-n-octylamine, 1. However, all the aggregates decreased in average size on extraction of uranium, the uranium-amine-sulfate complex apparently being entirely monomeric.

The exceptionally high uranium extraction power of N-benzyl-(branched secondary alkyl)amines (ORNL-1886) gives usable extraction even from high sulfate solutions, where other amines are ineffectual. The uranium extraction coefficient with 0.1 M N-benzylheptadecylamine in kerosene from 4 M sulfate solutions was ~1000 when the sulfuric acid content was 0.25 M, was 40 at 1 M, and was 1 at 3 M. A third liquid phase separated when the sulfuric acid concentration was 4 M.

It has been shown that the flow capacity of gravity settlers as used in the Amex or Dapex processes can be increased by use of coalescer media such as Berl saddles, glass cloth, and Selas rings or by application of an electrical potential. (AEC Activity 5310)

Thermochemistry and Calorimetry. — The low-temperature calorimeter designed to utilize liquid helium has been successfully operated at liquid-helium temperatures to measure the heat capacity of an empty calorimeter. The liquid-helium economy of the cryostat was found to be very satisfactory in that only 2.5 to 3 liters of liquid helium was required for the low-temperature range of the run. (AEC Activity 5330)

Chemistry of Corrosion. — The reduction reaction of uranyl ion on the surface of passive stainless steel electrodes in a solution of 0.010 f H_2SO_4 + 0.090 f Na_2SO_4, saturated with oxygen at 67°C, has been examined potentiostatically at a number of potentials. In agreement with earlier studies at a lower temperature (44°C), the data demonstrate that the reduction reaction is first order with respect to the concentration of uranyl ions in solution over the whole potential and concentration range studied, and that each individual reduction act involves the transfer of two unit charges across the electrode-solution interphase. The rate of reduction of molecular oxygen at the interphase is markedly increased by the product of the reduction reaction of uranyl ion. The data are described accurately by an electrochemical kinetic treatment which allows the evaluation of all pertinent specific rate constants. (AEC Activity 5330)

Ion Exchange Studies. — A series of measurements of the heat of exchange of chloride and bromide ions in Dowex 2 of nominal 2, 4, 10, and 24% divinylbenzene (DVB) cross-linking has been completed. The standard heats of exchange

for bromide replacing chloride ion, ΔH^o, for the 4, 10, and 24% DVB preparations are about -1300 cal/mole. The 2% DVB anion exchanger showed a ΔH^o of about -1450 cal/mole and a significant variation of ΔH with the mole fraction of bromide ion in the exchanger. The standard entropies, ΔS^o, for the 2, 4, 10, and 24% DVB exchangers were 3.5, 2.6, 2.1, and -1.7 e.u., respectively. The standard free energy changes, ΔF^o, were -495, -590, -755, and -825 cal/mole. The ΔF^o values were computed from selectivity coefficient measurements reported earlier.

To obtain an external check on the calorimetric measurements of the heats of cation exchange, selectivity coefficients for the sodium-hydrogen exchange were measured at 19.7, 25.1, and 30.1°C as a function of the equivalent fraction of sodium ion in the resin, x_{Na^+}, for a nominal 16% DVB Dowex 50 exchanger. Heats of cation exchange, ΔH, were computed for various values of x_{Na^+} from the slopes of the plots of the logarithm of the selectivity coefficient against the reciprocal of the temperature. These derived values of ΔH agreed well with the heats of exchange found calorimetrically except at the highest x_{Na^+} values, where large errors occur in the selectivity measurements. (AEC Activity 5330)

Chemistry of Technetium. — In the extraction of Tc(VII) from 0.5 M H_2SO_4 with 1-hexanol, the logarithm of the extraction coefficient E_a^o varies linearly with 1/T from 0 to 35°C, and the apparent heat of extraction is -5800 cal/mole. For Tc(VII) extracted from 2 M HNO_3 with a 10% solution of trioctylphosphine oxide (TOPO) in a hydrocarbon diluent, the dependence is not linear. At 25°C the slope of the curve indicates a heat of -9200 cal/mole.

Perrhenyl chloride and bromide (ReO_3Cl and ReO_3Br), pertechnetyl chloride (TcO_3Cl), and permanganyl chloride (MnO_3Cl) were found to be extractable from 18 M H_2SO_4 with CCl_4, $CHCl_3$, or hexane.

The visible and ultraviolet absorption spectra of pertechnetate ion, of perrhenyl chloride and bromide, of pertechnetyl chloride, and of permanganyl chloride show a strikingly more detailed fine structure in organic solution than in aqueous solution. The ReO_3Cl spectrum, for example, shows six well-defined absorption maxima and two additional ones readily discernible. The energy separation of these bands is approximately 900 cm^{-1}, indicating that an absorption band should appear in the infrared at approximately 11 μ. The infrared spectrum of gaseous ReO_3Cl has been measured elsewhere and does indeed show a single strong absorption band at 10.5 μ.

The absorption spectra showed that the species in cyclohexane with 0.1 mole/liter of TOPO after extraction of Tc(VII) from 0.5 M H_2SO_4 and of Tc(IV) from 6 M HCl are TcO_4^- and $TcCl_6^{--}$, respectively. In a solution of Re(VII) in 18 M H_2SO_4 a species containing two atoms of chlorine per atom of rhenium exists, as shown by the variation of the spectrum with the ratio of the concentration of chloride to the concentration of rhenium. This species may be $ReO_3Cl_2^-$ or $ReO_3Cl \cdot HCl$. (AEC Activity 5330)

Characterization of Short-Lived Fission Products. — The nuclide 2.7-min Rb^{90}, prepared by electrostatic collection of recoil ions in the decay of 33-sec Kr^{90}, has been studied by single-crystal and coincidence techniques. The spectrum obtained with a 3 x 3 in. NaI single-crystal scintillation spectrometer was analyzed into gamma-ray components of energy 0.52, 0.62, 0.85, 1.08, 1.4, 1.7, 1.9, 2.2, 2.6, 2.8, 3.4, 4.1, 4.4, and 5.3 Mev. Coincident with the 0.85-Mev gamma-ray peak were gamma rays at 0.39, 0.53, 0.87, 1.08, 1.3, 1.4, 1.7 Mev, and possibly higher energy components. With an anthracene crystal scintillation spectrometer, the maximum energy beta-ray group was found at about 6 Mev.

Irradiations of 97.7% Ge^{74} with 14-Mev neutrons from the fast-neutron facility at the ORNL Biology Division produced a nuclide of 7.8 ± 0.2 min half life, which has been identified as Ga^{74} because it follows the chemistry of gallium and because its beta decay excites the well-known first excited state of Ge^{74} at 0.60 Mev. As a final confirmation, samples of natural germanium (36.5% Ge^{74}) and of germanium enriched to 97.7% in Ge^{74} were irradiated in the same neutron flux, and the yield of the 8-min activity was proportional to the isotopic enrichment. Preliminary gamma-ray spectra measured with a 3 x 3 in. NaI crystal spectrometer showed intense gamma rays of 0.60- and 2.3-Mev energy, as well as weak transitions of 0.30, 0.50, 0.88, 1.1, 1.5, 1.9, and 2.8 Mev. The beta-ray spectrum observed with an anthracene spectrometer was resolved into three groups with end points at 2.65 ± 0.05, 2.0 ± 0.1, and 1.1 ± 0.1 Mev. (AEC Activity 5330)

High-Temperature Aqueous Solution Chemistry. — Phase equilibrium data for the system $NiO-SO_3-H_2O$ were obtained from 150 to 300°C and from 1 to 10^{-3} m SO_3. These data indicate that $NiSO_4 \cdot H_2O$ is the stable phase at high concentrations of SO_3 and at the higher temperatures, whereas several basic nickel sulfates appear to exist as stable species in lower concentration ranges. The completed phase equilibrium data will be combined with data for other three-component systems such as $UO_3-SO_3-H_2O$ and $CuO-SO_3-H_2O$ in an attempt to correlate mutual solubility interactions in aqueous homogeneous reactor solutions.

The profile of the sharp, strong 0.98-μ absorption maximum of Np(V) in 0.1 M solutions of DNO_3 in D_2O was studied as a function of temperature from 25°C to above 250°C. The band, which is well resolved by the Cary model 14 spectrophotometer, is slightly asymmetric, the integrated absorbance of the long-wavelength side being 1 to 2% greater than that of the short-wavelength side, both at 25 and at 251°C. The position of the absorbance maximum shifts smoothly from 9797 Å at 25°C to 9741 Å at 251°C. The half-intensity band width shows a striking behavior with increasing temperature, first decreasing (12%) from 58 Å at 25°C to a minimum of 51 Å at about 175°C, then slowly increasing to 54 Å at 251°C. The product of the absorbance at the band maximum and the half-intensity band width (normalized for solvent background and density change) and decreases linearly (35%) as the temperature increases from 25 to 251°C.

From a chemical point of view the above observations suggest an effect of temperature upon the solvation sphere of the NpO_2^+ ion and a concomitant interaction of the NO_3^- ion present with the NpO_2^+-solvate dipole system.

Several weaker absorption bands belonging to Np(V) and Np(VI) ions were less critically observed in the initial experiments. The results do indicate clearly, however, that the effect of temperature upon the position, shape, and intensity of the absorption bands varies qualitatively as well as quantitatively for the Np(V) and Np(VI) ions. (AEC Activity 5330)

Chemical Separation of Isotopes. — As part of the program for calculating isotopic separation factors between various oxides of nitrogen, 5 ml of liquid N_2O_4 containing 98% N^{15} was prepared from enriched nitric acid produced by the Nitrox process. The Raman spectrum of this material is being investigated by using a low-temperature cell designed for the Cary model 81 Raman spectrophotometer.

The infrared spectra of $N^{14}OCl$ and $N^{15}OCl$ have been recorded in the region from 2 to 40 μ by using CaF_2, NaCl, KBr, and CsBr prisms. The three fundamental bands, as well as most of the overtone and combination bands, were observed and identified. The measured frequency shifts in the fundamental bands on isotopic substitution are presented below.

Vibration Mode	Frequency Shift (cm^{-1})
N—O stretch (ν_1)	30.9
N—Cl stretch (ν_2)	14.0
O—N—Cl bend (ν_3)	1.8

The separation of the P and R branches of the bands in both molecules was the same within the limits of the measurements and averaged 19.3 cm^{-1}. (AEC Activity 5340)

METALLURGY

High-Temperature Reactions of Metals and Ceramics. — A strong, well-resolved nuclear magnetic resonance has been measured for the molten LiF-NaF-KF eutectic and for molten SnF_2. These measurements demonstrate the practicability of magnetic-resonance measurements on fused flouride salts. Further studies are planned in order to determine whether expected differences in fluoride-ion bonding can be detected by means of magnetic-resonance measurements. (AEC Activity 5420)

PROGRAM 6000 — BIOLOGY AND MEDICINE

BIOLOGY

Pathology and Physiology. — Young adult mice of the RF, LAF_1, L, A, BALB/c, and (BALB/c x RF)F_1 strains were exposed to varying doses (0 to 500 r) of whole-body 250-kv-peak x radiation and were observed thereafter until natural death. Lymphomas were induced by x radiation in all strains except the L, the highest induction rates occurring in the RF and (BALB/c x RF)F_1 strains. Myeloid leukemias were induced in all strains, the highest induction rates occurring in the RF and (BALB/c x RF)F_1 strains. The degree of susceptibility to the induction of lymphoma bore no discernible relation to the degree of susceptibility to the induction of myeloid leukemia. The results of these experiments and of those reported in the literature indicate that susceptibility to the induction of myeloid leukemia and susceptibility to the induction of lymphoma are properties widely shared by mice of diverse genetic constitutions. The relatively high susceptibility of RF mice to the induction of both diseases is a peculiarity of this strain; however, it may be transmitted to the F_1 hybrid offspring (BALB/c x RF) of RF mice and those of a less susceptible strain (BALB/c). (AEC Activity 6130)

Biophysics. — An elementary course in radiation physics has been given by members of the Biophysics Group and E. B. Darden, Jr., of the Pathology-Physiology Group.

Repetition of the paramagnetic resonance measurements and recalculation of the data on the long-lived free radicals produced in dry amino acids by Co^{60} gamma rays yield the following absorbed energies in electron volts per free radical produced: glycine, 160; glycylglycine, 100; β-alanine, 1300; and leucine, 1200. The glycine absorption curve shows a central peak and two satellites, the glycylglycine shows two almost equal peaks, the β-alanine shows one broad peak which is probably the envelope of a series, and the leucine shows four unequal peaks. The glycine pattern shows definite changes in shape several months after irradiation, and all the leucine peaks decay by at least an order of magnitude within one month after irradiation.

Experiments on photoreactivation in vitro have been continued. Preliminary results indicate that catalase cannot be photoreactivated in extracts of *E. coli* B.

In cooperation with D. Schwartz of the Cytology and Genetics Section, a study is being made of an extremely unstable gene for kernel color in maize. Monolayers of pollen grains have been exposed to ultraviolet and photoreactivating light in an effort to determine (1) whether ultraviolet light causes the mutation and, if so, (2) what dose is required, (3) the relative effectiveness of 2600 and 2800 Å, and (4) whether the mutation production can be photoreactivated. The exposures have been completed, and analysis of the data awaits maturation of the kernels. (AEC Activity 6130)

Microbial Protection and Recovery. — A mutant of *Escherichia coli* (H_7) which requires hemin for synthesis of porphyrin enzymes has continued to supply important information concerning the role of hydrogen peroxide and

other oxidizing products of irradiated water. Such cells in their "catalase negative" state show a "prompt" inactivation (during irradiation) and a "delayed" inactivation which is due to "sensitization" by the radiation and interaction of the sensitized cell with extracellularly produced H_2O_2. The mechanism of sensitization is being studied, and preliminary experiments indicate the possibility of dissociation of sensitization from the delayed inactivation.

It has been suggested that respiratory systems in living cells are linked to the oxygen effect. By use of the hemin-requiring cells it has been possible to show that the role of oxygen during irradiation is independent of the presence of a porphyrin-containing respiratory system in E. coli.

Previous experiments of this group showed that anaerobiosis during growth of E. coli (B/r) had a sizable effect on the inherent radiosensitivity of the cells. By use of the hemin-requiring mutant it has been possible to demonstrate that the inherent radioresistance of anaerobically cultured cells may be linked to porphyrin synthesis. The role of porphyrins in the radioresistant state is being investigated. (AEC Activity 6230)

Mammalian Recovery. — A colorimetric method has been developed by which a determination of the percentage of rat erythrocytes in the blood of a rat-mouse chimera can be made. This method takes advantage of the fact that mouse erythrocytes are hemolyzed by mannitol, whereas rat erythrocytes are not. The method has been checked successfully against standard agglutination tests.

Preliminary results indicate that red blood cell repopulation in the homologous situation ($LAF_1 \rightarrow C3H \times 101\ F_1$) is depressed by red blood cell plethora of the bone marrow donor.

Lethally irradiated $(L \times A)F_1$ mice were treated with intravenous injections of fetal and newborn liver or spleen tissue from homologous $(101 \times C3H)F_2$ or $(C3H \times 101)F_2$ donor mice. At 90 days the homologous recipients showed mortality of 75% in control-adult bone-marrow-treated mice, 40 to 50% mortality in mice receiving newborn liver or spleen suspension, and 30% mortality in mice receiving suspensions of fetal liver. In $(101 \times C3H)F_1$ mice treated with heterologous (rat) fetal or newborn liver and spleen suspensions after lethal irradiation, their 90-day mortality was not appreciably lowered from that of the rat-bone-marrow-treated controls. (AEC Activity 6230)

Radiation Immunology. — Newborn dba/2 mice, injected intracranially with C57BL/6 bone marrow, have not been found to be tolerant to skin grafts from the donor strain, when tested at eight weeks of age. Similarly, newborn C3H x 101 mice injected intracranially with LAF bone marrow, and used as either donors or recipients in radiation—bone marrow experiments with LAF mice at 12 weeks of age, show no evidence of a reduction in severity of the delayed death pattern characteristic of homologous bone marrow transplantation. On the contrary, there is some indication that the neonatal injections have had a sensitizing effect, particularly when the treated animals are the hosts in the bone marrow experiments. Preliminary evidence suggests that,

as in heterologous bone marrow experiments, homologous cytoplasmic particles as well as nuclear materials can presensitize the host to produce earlier and more extreme "delayed death patterns."

Tests for natural antibodies to sheep, rabbit, and rat red blood cell antigens in normal serum of mice of varying age groups show an earlier appearance, greater frequency, and greater titer of antibodies to the more distantly related antigen, that is, sheep >rabbit >rat. Preliminary studies on immune antibodies to the same antigens in two- to four-week-old mice show a differential response in favor of the more distantly related antigens.

Foreign bone marrow persists in normal mice for only one day. Minimum x-ray doses of 500, 950, 1150, and 1500 r, respectively, were found to be necessary to permit persistence of rat, hamster, guinea pig, and rabbit bone marrow for more than three days. (AEC Activity 6230)

PROGRAM 8000 — WORK FOR OTHERS

Oak Ridge School of Reactor Technology. — Seventy-six students completed the first session of the ORSORT-University Cooperative Program on February 22, 1958. On March 10, sixty-three students started the ORSORT phase of the second cooperative session. (AEC Activity 8712)

MISCELLANEOUS REPORTS

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VISITORS

FOREIGN VISITORS:

Bacq, Z. M., University of Liege, Belgium
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Buckingham, M. J., Duke University (Citizen of Australia)
Carr, M., Yale University (Citizen of Great Britain)
De Vathaire, F., French AEC, Paris, France
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